The Essential Oils

BY

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VOLUME FOUR

INDIVIDUAL ESSENTIAL OILS OF THE PLANT FAMILIES *GRAMINEAE, LAURACEAE, BURSERACEAE, MYRTACEAE, UMBELLIFERAE* AND *GERANIACEAE*

1950

D. VAN NOSTRAND COMPANY, INC.

TORONTO NEW YORK LONDON

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NEW YORK
D. Van Nostrand Company, Inc., 250 Fourth Avenue, New York 3
TORONTO
D. Van Nostrand Company (Canada), Ltd., 228 Bloor Street, Toronto
LONDON
Macmillan & Company, Ltd., St. Martin's Street, London, W.C. 2

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Published simultaneously in Canada by
D. VAN NOSTRAND COMPANY (Canada), LTD.

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Dedicated
to Mr. Frederick H. Leonhardt,
President of
Fritzsche Brothers, Inc.,
whose vision and generosity made this
work possible

PREFACE

Following the principle established in Vol. III of this series, the present volume consists of monographs on individual essential oils, arranged according to the botanical family of the plants from which they are derived. The important oils from six families are included. The author has again purposely avoided adherence to any strict taxonomic system in the sequence of these plant families, believing that the order followed here allows for greater flexibility in the practical use of the work by the essential oil producer, chemist and dealer. However, at the end of the final volume of the series there will be a table or key showing the classification of the essential oil plants according to a modern botanical system.

ERNEST GUENTHER

New York, N. Y.
September, 1950

NOTE

All temperatures given in this work are expressed in degrees Centigrade unless otherwise specified in the text.

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ACKNOWLEDGMENT

The collection of much of the material presented in this volume has been rendered difficult by the same problems of which the author wrote in the Preface to the preceding volume of this series. Many parts of the manuscript have required several rewritings, to keep pace with changing economic and political conditions throughout the world. In this tedious—but essential—task the author has had the generous assistance of a great number of planters, producers, government officials and researchers. To all of these collaborators and friends, the author wishes to express his deepest gratitude, regretting only that it is not feasible here to thank all of them individually. However, to the following the author feels a special indebtedness:

Dr. Teikichi Hiraizumi, President of Takasago Chemical Industry Co., Ltd., Tokyo, Japan, for his monographs on Japanese, Formosan and Chinese Camphor and Camphor Oils—monographs particularly valuable in that they were written by one of the foremost men in the field, and represent the first detailed treatment (at least in Western literature) of a complicated and obscure subject. In this work Dr. Hiraizumi had the benefit of the advice of Dr. Tessaku Ikeda, Director of the Association of Camphor Industry Engineers of Japan, Dr. Tsutomu Naito, Chemist, Takasago Chemical Industry Co., Ltd., Tokyo, and Mr. Tatsuo Tejima, formerly Forestry Engineer with the Government General of Formosa.

Mr. A. R. Penfold, Director of The Museum of Technology and Applied Science, Sydney, Australia, and Mr. F. R. Morrison, Economic Chemist of the same Institution, for their monographs on Australian Eucalyptus and Tea Tree Oils—subjects on which they are unchallenged authorities. Mr. Penfold and Mr. Morrison have been most ably assisted by Mr. H. H. G. McKern, Assistant Chemist, and Mr. J. L. Willis, Botanical Research Officer, also of The Museum of Technology and Applied Science, Sydney.

Dr. Kenneth A. Bartlett, United States Department of Agriculture, Mayaguez, Puerto Rico.

Mr. Ramon Bordas, Destilaciones Bordas Chinchurreta, S. A., Sevilla, Spain.

Mr. W. K. Burnside, W. K. Burnside Pty. Ltd., Melbourne, Australia.

Mr. Pierre Chauvet, Pierre Chauvet & Co., Seillans, Var, France.

Mr. Gerard J. Danco, Gerard J. Danco, Inc., New York, N. Y.

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Senator Louis Dejoie, Établissements Agricoles et Industriels Louis Dejoie, Port-au-Prince, Haiti, W. I.

Mr. João Dierberger, Dierberger Agro-Comercial Ltda., São Paulo, Brazil.

Mr. Jean Duclos, Les Fils de Maurice Duclos, Paris, France.

Dr. V. C. Dunlap, United Fruit Company, La Lima, Honduras, C. A.

Mr. Robert Garnier, Aroma, Fontenay-Sous-Bois, France.

Mr. G. O. Krauch, F. Krauch & Cia., S. A., Asunción, Paraguay.

Mr. F. J. Ippisch, Oficina Controladora de Aceites Esenciales, Guatemala, C. A.

Dr. Arnaud J. Loustalot, United States Department of Agriculture, Mayaguez, Puerto Rico.

Mr. Heinz Messtorff, Empresa Importadora, S. A., Mexico, D. F.

Dr. Yves-René Naves, Research Laboratories of Givaudan & Cie., Geneva, Switzerland.

Dr. J. A. Nijholt, Laboratorium Voor Scheikundig Onderzoek, Buitenzorg, Java.

Dr. E. O. Reif, Tela Railroad Company, La Lima, Honduras, C. A.

Mr. Carlos Schaeuffler, Schaeuffler Hermanos, Retalhuleu, Guatemala, C. A.

Mr. Edwin H. Sennhauser, Volkart Brothers, Inc., New York, N. Y.

Mr. R. O. Williams, Clove Growers Association, Zanzibar.

Much of the experimental work on the production of high-grade essential oils was carried out in Seillans (Var), France, in close collaboration with the author's good friend and associate for many years, Mr. Pierre Chauvet.

The Staff of the libraries of the Chemists' Club and of the Bronx Botanical Garden, both in New York, have been unfailingly courteous and helpful with this, as well as with all preceding volumes in this series.

The author is, as usual, deeply indebted to a number of his associates at Fritzsche Brothers, Inc., New York. Mr. B. F. Zimmer deserves particular mention for his great help in the evaluation of data on the physicochemical properties of essential oils examined in the laboratories of Fritzsche Brothers, Inc., over an extended period. Sincere thanks are due also to Mr. Edward E. Langenau, Dr. Darrell Althausen, Mr. W. P. Leidy and Mr. A. H. Hansen, Jr., of the same Company, for their constant advice and help with many phases of the work. And the author wants, once again, to express his kindest gratitude to his faithful secretarial staff—Mrs. Ann Blake Hencken, Miss Catherine McGuire, Mrs. Agnes Clancy Melody, and Mrs. Elizabeth Adelmann, who shouldered so much of the burden of a difficult manuscript.

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CHAPTER I

ESSENTIAL OILS OF THE PLANT FAMILY *GRAMINEAE*

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OIL OF PALMAROSA AND OIL OF GINGERGRASS

Introduction, Botany and Occurrence.—The grass *Cymbopogon martini* Stapf (syn. *Andropogon martini* Roxb.), in India commonly called "Rusha," "Rosha," or "Rusa" grass, occurs in two varieties: viz., *motia* and *sofia*. On distillation, the leaves and overground parts of the var. *motia* yield the oil of palmarosa which is of better quality and commercially much more important than the oil of gingergrass derived from the var. *sofia*.

Comparing single specimens of dried plants, Stapf [1] was unable to note any morphological difference between the two varieties. According to the observations of Burkill,[2] however, the two varieties do exhibit considerable differences: (1) as regards their field habitat (choice of altitude, soil, locality, growth—see below), and (2) chiefly in regard to the essential oil, which differs substantially in the two plants, the oil derived from the var. *motia* exhibiting a much finer odor than the oil from the var. *sofia*. Burkill also found grasses which were probably hybrids between *motia* and *sofia*.

Both varieties grow wild and abundantly in many parts of India (excepting deserts and the plains of the Punjab) from the Ganges River to the frontier of Afghanistan, and from the subtropical slopes of the Himalaya Mountains south to the 12th degree of latitude. From the commercial point of view the most important regions in which "Rosha" grass is processed for the oil are the Central Provinces, Berar, Khandesh, and Hyderabad (Deccan).

When collecting the grass, the native oil producers usually experience no difficulty in distinguishing between the two varieties, as they seldom grow side by side, but in separate stands at different altitudes and in different environments.

Motia seems to prefer a dry, well-drained soil of basaltic composition, relatively bare and sunny mountain slopes with southern exposure, open forest, or sunny clearings on sparsely wooded land. It thrives in regions with an annual rainfall ranging from 32 to 35 in., but does not withstand stagnant water. *Sofia*, on the other hand, grows best at lower altitudes in moist and poorly drained soil, in valleys, or in dense forests kept humid by rain, dew, and fog. *Motia* usually occurs in scattered patches consisting of stocks 1 or 2 ft. apart, whereas *sofia* grows abundantly and densely, and

[1] *Ber. Schimmel & Co.*, April (1907), 50.

[2] "First Notes on *Cymbopogon Martini* Stapf," *J. Proc. Asiatic Soc. Bengal* **5**, No. 3 (1909). Cf. *Ber. Schimmel & Co.*, October (1909), 85.

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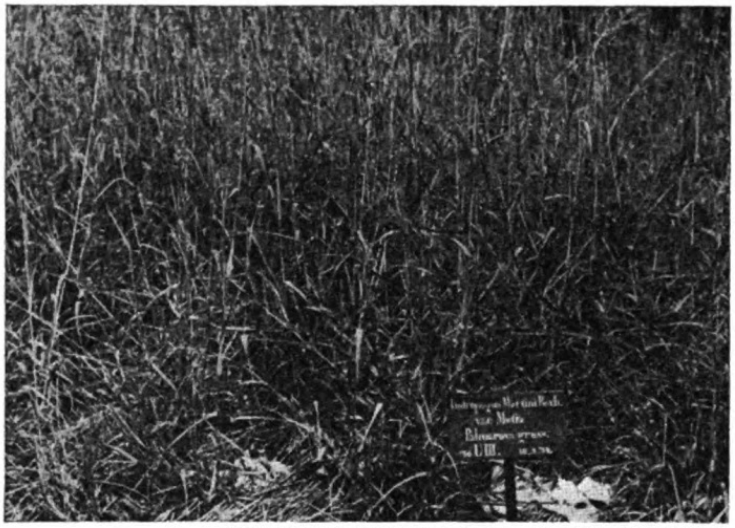

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FIG. 1. Production of palmarosa oil in India. View of palmarosa grass.

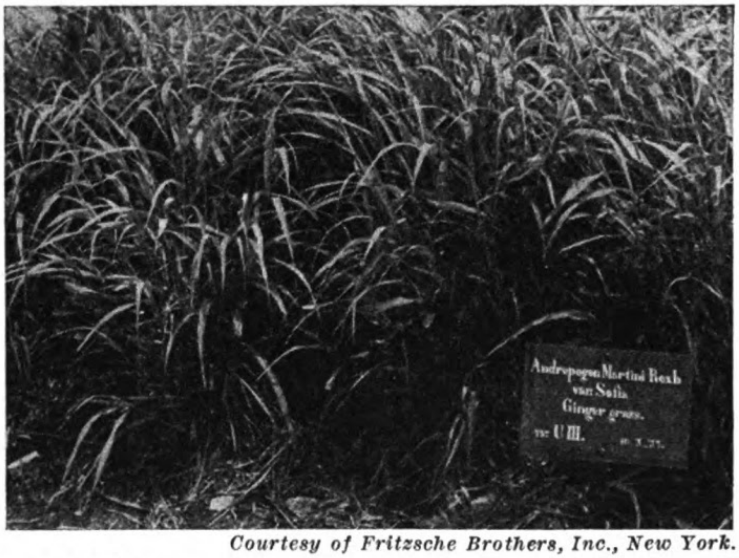

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FIG. 2. Production of gingergrass oil in India. View of gingergrass.

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frequently covers large areas. Unlike *motia,* the var. *sofia* is characterized by a large number of radical leaves which form a different angle with the sheath than do the leaves of the var. *motia.*

OIL OF PALMAROSA
(East Indian Geranium Oil)

Essence de Palmarosa *Aceite Esencial Palmarosa (Geranio de Turquia)*
Palmarosaöl *Oleum Palmarosae (Geranii Indicum)*

Oil of Palmarosa India

The old term "Indian" or "Turkish" geranium oil, which formerly was occasionally applied to palmarosa oil, dates back to the time when the oil was shipped from Bombay to ports of the Red Sea and transported, partly by land, to Constantinople and Bulgaria, where the oil was often used for the adulteration of rose oil.

In India, where the bulk of commercial palmarosa oil originates, the oil is distilled from *Cymbopogon martini* Stapf var. *motia* (syn. *Andropogon martini* Roxb. var. *motia*) which grass grows wild in several parts of the subcontinent. (Efforts to raise the grass on plantations have been made in the Seychelles Islands and in Java—cf. the monographs on "Oil of Palmarosa Java" and "Oil of Palmarosa Seychelles.")

As was mentioned above, in most producing regions of India, the palmarosa grass and the closely related gingergrass do not grow side by side. However, in a few sections the two varieties occur in neighboring places which, obviously, tempts the native cutters and oil producers to collect the grass from both varieties, and to distill these together. Oils distilled from mixed *motia* and *sofia* grass are, of course, of subnormal quality, but fortunately in most regions the cutters and distillers exercise care in the selection of the distillation material and produce palmarosa oil exclusively.

Producing Regions.—The principal producing regions of palmarosa oil in India, listed according to quality and quantity of the oil, are:

1. Berar and Central Provinces; in the districts of Akola, Elichpur, Amraoti, and Nagpur. Much of the oil comes from the slopes and highlands of the Melghat. Principal marketing center is the town of Elichpur.

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The oil produced in the province of Berar has usually been of best quality, possessing a sweet rosy note and a high total alcohol (geraniol) content.

2. Khandesh districts (northeast of Bombay), particularly in the vicinity of Pimpalner, Akrani, and Nandurbar. This type of oil has a somewhat lower total alcohol (geraniol) content, but is still true palmarosa oil.

3. Hyderabad (Deccan), particularly in Zaheerabad and Mahbubnagar. Here the oil is distilled from both the var. *motia* and var. *sofia*, the latter prevailing. The oil originating from Hyderabad is therefore mostly gingergrass oil.

4. Madras, Punjab and Bengal produce almost exclusively gingergrass oil.

The producing areas are partly wilderness and remote from modern civilization. Production of the oil is, therefore, carried out by natives under utterly primitive conditions.

Collection of the Grass and Economic Setup.—As was pointed out above, in India palmarosa is not planted on an organized basis, as it grows wild over large tracts of land. On privately owned land there are no restrictions against cutting and distillation of the grass. However, much of the land is government property, in which case the licenses for cutting and distilling are auctioned off to the highest bidding parties, which also must furnish guarantee that they will actually cut the grass. Palmarosa and gingergrass have a tendency to spread and to invade arable land and pastures, making them unfit for cattle, which cannot feed on these highly aromatic plants. In view of these circumstances the government does not readily grant cutting licenses to outsiders—merchants and oil dealers in Bombay, for example—because such parties cannot always offer sufficient guarantee that the plants will actually be cut.

To overcome government objections the merchants or exporters usually operate through middlemen or village agents known both to the government and to the native distillers. Provided with funds the middlemen in the villages of the producing regions then procure the necessary distilling equipment and hire crews of cutters. Actual distillation is carried out by members of certain native tribes or castes who have practiced this craft for generations. The distillers work for the agent on a sharecropping basis, retaining about one-quarter of the oil produced, while the agent receives three-quarters. The distillers are free to sell their share to whomever they please, but they usually prefer selling to the agent, the price being determined by prevailing market quotations in Bombay. Much oriental shrewdness is displayed in this sort of trading.

The di

in Bombay, the oil often remains the property of the agent in the village; the Bombay exporters or money lenders then purchase the lot only when they receive definite export orders from abroad.

Distillation and Yield of Oil.—Distillation of the grass is carried out between October and December or January, depending upon the rainfall and the development of the plants. The leaves and the upper third of the stems are cut and tied into bundles of ½ lb. each. Systematic experiments by Lall [1] have shown that the leaves of palmarosa grass (*motia*) yield the maximum amount of oil (1.39 per cent [2]) in October, when the plants begin to bloom. After the flowering period the oil content of the plant diminishes steadily, declining to the minimum (0.77 per cent [2]) in the beginning of March. The highest yield of oil is obtained from fully dried grass one week after it has been cut. The top of the grass seems to contain most of the oil, but it is not advisable to cut only the tops for distillation, because the grass will then exhaust itself by too rapid regrowth. In other words, the plant will grow up again within a month but will contain much less essential oil. In actual practice, the natives cut the grass quite close to the ground, in order to gather as much plant material as possible (by weight). Lall's work [3] further indicates that the (lower) stalks of the plant contain only traces of oil; stalks and flowers yield an oil of subnormal quality.

According to Narian and Das Gupta [4] the yield of oil from fresh or carefully dried plant material may amount to 1 per cent; if dried carelessly the yield may be as low as 0.15 per cent. The yield ranges from 1 to 1.25 per cent, if the distillation is carried out with absolutely fresh grass immediately after cutting. The yields from each portion of the plant, when distilled separately, are as follows:

Stalks	0.04 per cent of oil
Leaves	1.32 per cent of oil
Flower Heads	1.71 per cent of oil

Rakshit and Dutt [5] reported these yields of oil:

Whole Plants (before Flowering Tops Appear)	0.13 to 0.21%
Flowering Tops (Separately)	0.70 to 0.98%
Lower Portions	0.39 to 0.61%

Rakshit and Dutt,[6] however, did not indicate the moisture content of the plant material at the time it was distilled; therefore it is not clear whether their yields are based on fresh grass, air-dried grass, or moisture-free grass.

[1] *Indian J. Agr. Science* **5** (1935), 415. *Bull. Imp. Inst.* **33** (1935), 472.
[2] In the opinion of the author, these figures are extremely high.
[3] *Indian J. Agr. Science* **5** (1935), 415. *Bull. Imp. Inst.* **33** (1935), 472.
[4] *Indian Soap J.* **14** (1948). 260.
[5] *Ibid.* **13**, August (1947), 27.
[6] *Ibid.*

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This pipe is tied to the still head with palm-leaf rope or some other simple material. The long arm of the bamboo pipe, wrapped with cord, reaches into a pot or vessel serving as condenser (and simultaneously as Florentine flask). This receiver, immersed up to its neck in water, is supported by a wooden frame beneath. The top of the receiving vessel, into which the bamboo pipe reaches, is covered with burlap. Wood cut in the nearby forests serves as fuel for the open fire.

The still content is brought to a boil, and essential oil vapors and distillation water condense in the partly immersed receiving vessel. Distillation lasts from 4 to 5 hr., including the time required (about 1½ hr.) to heat the water in the still to the boiling point. Distillation completed, the oil collected in the receiver is usually skimmed off the distillation water. Frequently, however, oil and water are not separated on the spot; the whole mixture is simply poured into galvanized iron containers of 2 to 4 gal. capacity. These vessels, containing water and oil, are then carried overland to the nearest village, where an expert, or an agent for a Bombay exporter, separates the oil from the water by simple decantation. The oil is finally filtered through cloth into containers or drums. These drums, usually holding 45 gal., serve for storage of the oil until the end of the harvest, or until the filled drums can be shipped to Bombay.

If oil and water are not immediately separated near the stills, the oil stays in contact with water for several days, which obviously does not improve its quality. In general, the oil remains in iron drums for weeks or months, exposed at times to the heat of India's climate. Such exposure, together with the presence of a little water in the oil, is not conducive to good quality.

The yield of oil could undoubtedly be raised substantially if distillation were carried out in more efficient stills. In fact, Pearson [8] proved, by experiments carried out in 1915, that distillation of palmarosa grass in direct steam stills increased the yield of oil 20 to 100 per cent. (According to Trotter,[9] the crude old "country" stills in a few places have been replaced with regular steam stills in which distillation is carried out with steam generated in a separate boiler.)

In general, the quality of the oil suffers from the crude methods of production and from the direct contact of plants with the walls of the vessels heated by open fire, as well as from the inefficent method of condensation. The relatively low ester content of the Indian palmarosa oils, as compared with that of the Java type, is probably the result of these conditions. Since the plant material is immersed in boiling water during the distillation the greater part of the esters will be hydrolyzed (see Vol. I of this work, pp. 112, 120, 142, and 145).

[8] *Indian F*

Shipping of the Oil.—Exporters in Bombay leave the oil in the drums until they receive shipping orders from abroad. The lot is then filtered through paper, or (more often) through cloth, and filled into drums or special containers for export. Years ago these containers consisted of copper, heavily tinned on both sides, and protected on the outside by a net of heavy cord. They were made in Bombay and were quite expensive. More recently the oil has been shipped in ordinary galvanized iron drums.

Physicochemical Properties.—According to Gildemeister and Hoffmann,[10] the properties of Indian palmarosa oil vary within the following limits:

Specific Gravity at 15°	0.887 to 0.900
Optical Rotation	+6° 0′ to −3° 0′ (usually +1° 0′ to −2° 0′)
Refractive Index at 20°	1.472 to 1.477
Acid Number	0.5 to 3.0
Ester Number	12 to 48
Ester Number after Acetylation	226 to 274
Total Alcohol Content, Calculated as Geraniol	74.8 to 94.8%
Solubility	Soluble in 1.5 to 3 and more vol. of 70% alcohol, in very rare cases with opalescence to turbidity. Oils of high geraniol content are soluble in 3 to 5 vol. of 60% alcohol and more

Shipments of Indian palmarosa oil received and examined by Fritzsche Brothers, Inc., New York, had properties varying within these limits:

Specific Gravity at 15°/15°	0.887 to 0.895
Optical Rotation	−1° 34′ to +2° 45′
Refractive Index at 20°	1.4730 to 1.4760
Acid Number	0.7 to 1.1
Ester Content, Calculated as Geranyl Acetate	3.3 to 12.6%
Total Alcohol Content, Calculated as Geraniol	84 to 94%
Solubility	Soluble in 3 to 4 vol. and more of 60% alcohol

Narian and Das Gupta [11] reported the following properties of pure palmarosa oil (var. *motia*) from India:

Specific Gravity	0.886 to 0.889
Optical Rotation	−3° 0′ to +5° 0′
Refractive Index	1.4720 to 1.4780
Acid Number	Up to 3
Ester Number	12 to 50
Total Geraniol Content	78 to 94%
Solubility in 70% Alcohol	Soluble in 1 to 3 vol.

[10] "Die Ätherischen Öle," 3d Ed., Vol. II, 294.
[11] *Indian Soap J.* **14** (1948), 260.

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According to the same authors,[12] the most characteristic factors in the evaluation of a palmarosa oil (var. *motia*) are its sweet rose-like odor, and solubility in 60 per cent alcohol. If an oil is soluble in 2.2 to 4.2 volumes of 60 per cent alcohol, it is likely that it contains a high percentage of free geraniol.

The characteristic feature of palmarosa oil (var. *motia*) from Hyderabad is that it contains a much higher percentage of esters (11 to almost 15 per cent, calculated as geranyl acetate) than the oils from the Central Provinces (4.33 to 7.14 per cent of esters). Furthermore, the oils from Hyderabad are occasionally insoluble in up to 10 volumes of 60 per cent alcohol.

The Hyderabad oils also differ in themselves to a certain extent, the quality depending upon their origin. A sample of palmarosa oil (var. *motia*) [13] from Hyderabad showed a low geraniol content and was insoluble in 70 per cent alcohol up to 12 volumes. There was no adulteration found in the sample, but the oil contained a relatively high percentage of terpenes. The grass used for distillation of this oil was said to be of inferior quality (probably var. *sofia*—the author).

On the basis of their investigation, Narian and Das Gupta [14] arrived at the conclusion that palmarosa oils from Amraoti (C.P.) and Hyderabad have these properties:

	Amraoti	*Hyderabad*
Specific Gravity	0.887 to 0.892	0.891 to 0.900
Refractive Index at 20°	1.4685 to 1.4729	1.4723 to 1.4763
Acid Number	Up to 1.6	Up to 1.8
Total Free Alcohol Content, Calculated as Geraniol	80%	70%
Solubility in 70% Alcohol	Soluble in 1 to 3 vol.	Soluble in 1 to 3 vol.
Solubility in 60% Alcohol	Soluble in 1 to 4 vol.	Soluble in 1 to 4.5 vol.

Adulteration.—The most common adulterant of palmarosa oil is the inferior gingergrass oil, which may be recognized by an abnormally low total geraniol content in the suspected oil. The total geraniol content can be assayed by the usual method of acetylation, and saponification of the acetylized oil (see Vol. I of this work, p. 271). Oils containing less than 75 per cent of total geraniol should be rejected, the highest qualities of palmarosa oil assaying from 85 to 90 per cent or even more.

Other adulterants used by the native distillers include coconut oil, which can be isolated by freezing (cf. Vol. I of this work, p. 344), and turpentine

[12] *Ibid.*, 263.

[13] In the author's opinion the oils from Hyderabad are probably not distilled exclusively from the var. *motia*, but from var. *sofia* (gingergrass) or from hybrids of the two varieties. Their properties approach those of gingergrass oil.

[14] *Indian Soap J.* **14** (1948), 263.

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oil, the presence of which is indicated by a lowered solubility in 70 per cent alcohol (cf. Vol. I of this work, p. 337). Kerosene and mineral oils are also employed. These can be identified by fractionation and by the so-called oleum test described in Vol. I of this work, p. 332.

Native dealers evaluate an oil simply by shaking it in a small bottle or by hitting the bottle against the palm of the hand. If the air bubbles which then rise to the surface disappear quickly, the oil is assumed to be normal. If they remain a few seconds, and foam forms, the oil is suspect. Exporters in Bombay evaluate palmarosa oil by a simple solubility test, a very good solubility indicating a high geraniol content and hence a good quality.

Total Production and Exports.—The yearly total production of palmarosa oil in India ranges from 20,000 to 40,000 lb. Export figures [15] from Bombay for the years prior to World War II were as follows:

	Pounds
1935	32,083
1936	26,550
1937	35,316
1938	27,380

Of the latter quantity

21,805 lb. went to Europe
5,200 lb. went to America
375 lb. went to Japan

More recent exports [16] of palmarosa oil from India have been as follows (for the fiscal year ending March 31st):

	Gallons
1939–1940	3,568
1940–1941	3,520
1941–1942	7,421
1942–1943	4,345
1943–1944	360
1944–1945	7,061
1945–1946	9,293

Oil of Palmarosa Seychelles

For a number of years efforts have been made in the Seychelles Islands to produce a high-grade palmarosa oil. For this purpose *Cymbopogon martini* Stapf var. *motia* was planted exclusively and all gingergrass (var. *sofia*)

[15] *Ber. Schimmel & Co.* (1939), 60.

[16] "Accounts Relating to the Sea-borne Trade and Navigation of British India," *India Commercial Intelligence Statistics Dept.*, March (1946).

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eradicated. According to trade news reports,[17] in the Seychelles palmarosa grows exceptionally well on the sandy coral plateaus. The grass is easily propagated by means of soft cuttings rooted in sand under shade.

The Imperial Institute [18] in London reported properties for two oils from the Seychelles Islands, which resembled those of the Indian oils. The total alcohol content (calculated as geraniol) was 90.1 per cent. The odor of the Seychelles oils was somewhat softer than that of the Indian oils.

Oil of Palmarosa Java

A few years prior to the outbreak of World War II, Java began to produce oil of palmarosa from *Cymbopogon martini* Stapf var. *motia* (syn. *Andropogon martini* Roxb. var. *motia*), the true palmarosa. Originally the quantities were very small, but in 1937 Java exported 2,755 kg. of oil, chiefly to Holland and England. In 1938, 4,721 kg. were exported.

Java palmarosa oil is distilled exclusively from planted and cultivated grass. Before World War II there were in Java five producers, all European planters, the most important one distilling yearly from 1,000 to 2,000 kg. of oil in Padalarang, near Bandoeng, and another planter somewhat smaller quantities in Modjo-Agoeng near Sourabaya, eastern Java.

The grass, which was originally introduced from India, is planted, by root division, during the rainy season. The segments are set out 80 cm. by 80 cm. apart. When the plants have grown to a height of about 2.25 m. they are ready for cutting. Harvesting can be repeated every four months, but after five years the planting must be renewed. As was said above, in 1939 all palmarosa plantations in Java were in the hands of European producers, the many native and Chinese growers of citronella not yet having started with palmarosa. All palmarosa plantings were, therefore, properly taken care of, carefully weeded out, and in excellent condition. This might be one of the reasons for the good quality of the Java oil.

Distillation was carried out by modern methods. The above mentioned producer in Padalarang, for example, employed direct steam stills of about 5 cu. m. capacity. Distilling one charge of 600 kg. of grass with live steam for 3 hr., he obtained about 1 kg. of oil. The oil yield, therefore, averaged 0.17 per cent.

Distillation with indirect steam and cohobation of the distillation waters also produced a very high grade of palmarosa oil.

Quality of the Java Oil.—It might be interesting in this connection to comment on the quality of the Java oil. Compared with the Indian palmarosa oil, the Java product should be of superior quality because:

[17] *Perfumery Essential Oil Record* **30** (1939), 42; **31** (1940), 63.
[18] *Bull. Imp. Inst.* **25** (1927), 112.

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1. In Java all palmarosa grass originates from well-kept plantations, confined to *Cymbopogon martini* Stapf var. *motia*. In India, on the other hand, the plant material consists of wild growing grass, occasionally mixed with the inferior *sofia* variety (gingergrass).

2. Distillation in Java, as was mentioned above, is carried out in modern steam stills by large-scale producers,[19] whereas in India the grass is distilled by natives in primitive, direct fire stills with insufficient condensation. While the plant material is immersed in boiling water in the Indian stills, hydrolysis of esters takes place which results in a generally low ester content of the Indian oils. Direct steam distillation, as practiced in Java, results in oils with a much higher ester content. Moreover, the Indian oils are frequently improperly stored and may be exposed for weeks or months to the high temperature of central India.

3. In Java every single lot of oil must be analyzed in the Government Analytical Laboratories of Buitenzorg prior to the granting of an export license. Inferior oils are rejected.

In view of all these factors, it would appear that the Java oil should be of excellent quality, unless the plant itself, after its introduction from India, gradually degenerates, under the climatic and soil conditions of Java, or under cultivation.

The difference between the Indian and the Java palmarosa oil is, in point of fact, quite pronounced. Whether this results from the superior methods of production employed in Java, or from mutation of the plant in its new environment, is open to question. When, in 1937, commercial shipments of Java oil reached Europe, the consumers, much to the dismay of the Buitenzorg Experiment Station in Java, rejected the oil as being different from the usual commercial oil from India, and unsuitable for their purposes. Such critical judgment is often encountered in our industry when a new type of oil is introduced to replace an old one, to which the trade has become accustomed through many years of use.

In the author's opinion, the Java oil represents a high grade of palmarosa oil, superior, in fact, to the Indian product, which has a somewhat harsh by-note. Because of its soft, slightly fruity and rose-like character, the Java oil can be used in perfumes and cosmetics, whereas the stronger Indian oil should be employed for the scenting of soaps. The Java product may also serve as a good starting material for the extraction of a very fine geraniol. There is undoubtedly a place for both oils, but each type should be marketed

[19] At the time of this writing (1949), it is still difficult to judge to what extent conditions have changed during the Japanese occupation and the civil

under its proper label, as has been customary with several other essential oils—geranium oils from Réunion and Algeria, vetiver oils from Java and Réunion, and patchouly oils from Sumatra and Singapore, for example.

Physicochemical Properties.—According to Koolhaas,[20] the properties of Java palmarosa oil vary within the following limits:

Specific Gravity at 15°	0.886 to 0.899
Optical Rotation	−3° 0′ to +5° 0′
Refractive Index at 20°	1.4720 to 1.4780
Acid Number	0 to 3
Ester Number	12 to 50
Total Geraniol Content	78 to 94%
Aldehyde Content, Calculated as $C_{10}H_{18}O$ (Hydroxylamine Method)	0 to 10%
Solubility in 70% Alcohol	One volume is soluble in 1½ to 10 vol.

Shipments of Java palmarosa oil examined by Fritzsche Brothers, Inc., New York, had these properties:

Specific Gravity at 15°/15°	0.888 to 0.894
Optical Rotation	−0° 6′ to +0° 18′
Refractive Index at 20°	1.4729 to 1.4748
Acid Number	1.4 to 2.8
Ester Content, Calculated as Geranyl Acetate	9.5 to 17.6%
Total Alcohol Content, Calculated as Geraniol	85.7 to 90.2%
Aldehyde Content, Calculated as Citronellal (Hydroxylamine Hydrochloride Method)	1.2 to 10.8%
Solubility at 20°	Occasionally soluble in 3.5 vol. of 60% alcohol. Usually soluble in 2 vol of 70% alcohol, opalescent in 4.5 vol. and more

These limits may be somewhat too narrow; only few commercial lots have been available and more data are required before definite limits can be established.

Adulteration.—The careful examination by the Buitenzorg Government Laboratories to which each shipment of Java palmarosa oil is subjected prevents the exportation of adulterated oils.

Chemical Composition of Palmarosa Oil

The first research on the chemical composition of palmarosa oil derived from true *Cymbopogon martini* Stapf var. *motia* was carried out by Jacobsen [21] who identified an alcohol $C_{10}H_{18}O$ as main constituent and assigned

[20] Private communication of Dr. D. R. Koolhaas, Buitenzorg, Java (1939).
[21] *Liebigs Ann.* **157** (1871), 232.

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to it the name geraniol. Years later Semmler [22] confirmed the validity of the formula $C_{10}H_{18}O$ and found that this important terpene alcohol belongs to the aliphatic series.

More recently van Hulssen, Koolhaas, and Rowaan [23] studied the aldehydes present in palmarosa oil and arrived at the conclusion that the oils distilled in Java contain from 1.3 to 10.2 (on the average 7.0) per cent of aldehydes, whereas the oils produced in India (formerly British East India) contain 5.1 to 9.4 per cent [24] of aldehydes. The aldehydes of the Java oil consist mainly of citral, with occasionally some citronellal. The fresh odor occasionally noted in some lots of palmarosa oil is caused perhaps by low boiling aldehydes. The same authors also confirmed the presence of those constituents which had been identified in earlier investigations of palmarosa oil from India.

The following substances, listed approximately in order of their boiling points, have been observed in palmarosa oils from India and Java:

Formaldehyde. By van Hulssen, Koolhaas and Rowaan.[25]

Isovaleraldehyde. By the same authors.

Methyl Heptenone. Traces of this ketone in the Indian oil were noted by Schimmel & Co.[26] who prepared the semicarbazone m. 135°.

Dipentene. The same workers also found that the Indian oil contains about 1 per cent of dipentene, which was characterized by the preparation of its tetrabromide m. 125°, and nitrolbenzylamine m. 109°–110°.

Citronellal. Identified in a Java type of oil by van Hulssen, Koolhaas and Rowaan.[27] In the opinion of these authors, the presence of citronellal in one of their Java oils is perhaps due to the fact that this particular lot was distilled from a special horticultural variety of *Cymbopogon martini* Stapf var. *motia*.

Citral. Observed by the same workers [28] in palmarosa from Java and India. The aldehydes present in the Java type of oil consist mainly of citral.

Geraniol. When first isolating this main constituent from Indian palmarosa oil, Jacobsen [29] purified it through its calcium chloride addition compound, which procedure later became of great importance for the isolation (and purification) of primary terpene alcohols from complex mixtures.

[22] *Ber.* **23** (1890), 1098.
[23] *Indische Mercuur* **61** (1938), 489. Cf. *Mededeelingen van het Laborat. voor Scheikundig Onderzoek te Buitenzorg. Handelsmuseum van het Koloniaal Instituut* 1938.
[24] The laboratories of Fritzsche Brothers, Inc., New York, examined a number of pure Indian palmarosa oils which contained very little, if any, aldehydes.
[25] *Indische Mercuur* **61** (1938), 489. Cf. *Mededeelingen van het Laborat. voor Scheikundig Onderzoek te Buitenzorg. Handelsmuseum van het Koloniaal Instituut* 1938.
[26] *Ber. Schimmel & Co.*, April (1905), 39.
[27] *Indische Mercuur* **61** (1938), 489. Cf. *Mededeelingen van het Laborat. voor Scheikundig Onderzoek te Buitenzorg. Handelsmuseum van het Koloniaal Instituut* 1938.
[28] *Ibid.*
[29] *Liebigs Ann.* **157** (1871), 232.

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Indian oil of palmarosa contains from 75 to 95 per cent of (free and combined) geraniol. Van Hulssen, Koolhaas, and Rowaan [30] reported a geraniol content (free and combined) of 82–94 per cent, for the Java type of palmarosa oil.

Geranyl Acetate and Geranyl *n*-Caproate. According to Gildemeister and Stephan,[31] about 3 to 15 per cent of the geraniol occurring in Indian palmarosa oil is present in the form of acetic and *n*-caproic esters, the quantities of the two esters being approximately equal. They arrived at this conclusion by analyzing the silver salts of acetic and *n*-caproic acids.

Farnesol. Elze [32] reported on the occurrence of this sesquiterpene alcohol in Indian palmarosa oil.

Years ago Flatau and Labbé [33] claimed that palmarosa oil also contains citronellol and a fatty acid $C_{14}H_{28}O_2$, m. 28°. According to Schimmel & Co.,[34] however, this is not the case; the oil investigated by Flatau and Labbé was probably adulterated with coconut or some other fatty oil.

Investigating a palmarosa oil of Sukhum (U.S.S.R.) origin, Bulanova [35] found that the geraniol content of this type of oil is identical with that of the imported (Indian) type of oil. The Sukhum oil contained:

Geraniol	77.68%
Citronellol	1.18%
Aldehydes and Terpenes	Traces, not studied
Sesquiterpene Alcohols	About 3.9%

As regards the presence of citronellol in palmarosa oil (reported by Flatau and Labbé, and by Bulanova—see above), the chemists of Schimmel & Co. disproved this claim years ago. To eliminate any doubt, the laboratories of Fritzsche Brothers, Inc.,[36] more recently examined genuine East Indian as well as Java palmarosa oils and, using the (modified) method of Tiemann and Schmidt [37] for the separation of citronellol and geraniol, arrived at the conclusion that citronellol is not present, either free or as ester, in East Indian or Java palmarosa oils. Adulteration of palmarosa oil by the addition of "geraniol fractions" (which always contain some citronellol) can thus be detected.

In the course of their investigation the chemists of Fritzsche Brothers, Inc., also confirmed the presence of acetic acid and of *n*-caproic acid in East Indian and Java palmarosa oils.

[30] *Indische Mercuur* **61** (1938), 489. Cf. *Mededeelingen van het Laborat. voor Scheikundig Onderzoek te Buitenzorg. Handelsmuseum van het Koloniaal Instituut* 1938.
[31] *Arch. Pharm.* **234** (1896), 328.
[32] *Chem. Ztg.* **34** (1910), 857.
[33] *Compt. rend.* **126** (1898), 1725. *Bull. soc. chim.* [3], **19** (1898), 633.
[34] *Ber. Schimmel & Co.,* October (1898), 29.
[35] *Trudy Vsesoyuz. Inst. Efirno-Maslichnoi Prom.* **8** (1940), 183. *Chem. Abstracts* **37** (1943), 3558.
[36] Publication forthcoming.
[37] *Ber.* **29** (1896), 921.

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Recently Rakshit and Dutt [38] reported that Indian palmarosa oil contains from 0.04 to 6.0 per cent of phenols. Since this latter percentage seems rather high, the laboratories of Fritzsche Brothers, Inc., New York, examined a few lots of Indian palmarosa oil for the presence of phenols and found that they contained about 0.04 per cent of phenols.

Use.—At one time the oil was used as an adulterant of Bulgarian or Turkish rose oil. Today palmarosa oil is the source of a very high grade of geraniol. The oil is widely employed in cosmetics, particularly in soaps, to which it imparts a pronounced and lasting rose note. Geraniol, the chief constituent of palmarosa oil, being quite alkali resistant, the oil stands up well in soaps.

Another use of palmarosa oil is in the flavoring of tobacco.

OIL OF GINGERGRASS

As was pointed out in the introduction to the monograph on "Oil of Palmarosa," oil of gingergrass is distilled in India from the wild growing grass, *Cymbopogon martini* Stapf var. *sofia* (syn. *Andropogon martini* Roxb. var. *sofia*). Production regions include Hyderabad, parts of Bengal, Punjab, and the presidency of Madras. The oil originating from Madras is derived exclusively from gingergrass, and not partly from palmarosa, as may be the case in the other producing regions.

Harvesting of the plants, distillation and handling of the gingergrass oil take place under conditions almost identical with those described in the monograph on "Oil of Palmarosa."

The bulk of the oil is shipped to ports of the Red Sea, and to the Sudan, for use as a cheap perfume for the native population. In East Africa the oil is also employed, in conjunction with sandalwood oil, as a mosquito repellent that is claimed to be quite effective. Applied alone, the oil dries too rapidly, for which reason it should be incorporated into a base of fatty oils. Gingergrass oil is also said to have some value as a remedy for stiff joints, lumbago, and other ailments.

The export figures for gingergrass oil prior to World War II were as follows: [1]

[38] *Indian Soap J.* **13,** August (1947), **27.**
[1] *Ber. Schimmel & Co.* (1939), **36.**

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	Pounds
1934	63,529
1935	56,290
1936	60,250
1937	26,369
1938	61,445

Of the last quantity, 1,991 lb. were shipped to Europe; 59,454 lb. went to Red Sea ports.

As regards cultivation experiments (yield of plant material, yield of oil from various parts of the plant, etc.) recently carried out with gingergrass in India, see the monograph on "Oil of Palmarosa," section "Distillation and Yield of Oil"—above.

Physicochemical Properties.—According to Gildemeister and Hoffmann,[2] the properties of gingergrass oil vary within the following limits:

Specific Gravity at 15°	0.900 to 0.953
Optical Rotation	+54° 0′ to −30° 0′
Refractive Index at 20°	1.4780 to 1.4930
Acid Number	Up to 6.2
Ester Number	8 to 29; in one case 54.5
Ester Number after Acetylation	120 to 200
Total Alcohol Content, Calculated as Geraniol *	36.3 to 64.7%
Solubility	Mostly soluble in 2 to 3 vol. of 70% alcohol, opalescent to turbid with more alcohol. Soluble in 0.5 to 1.5 vol. of 80% alcohol and more, in rare cases with slight opalescence

* Calculated from the ester number after acetylation.

Data on the physicochemical properties of gingergrass oil reported recently in some Indian publications will not be cited here, as the total geraniol content of the oils described has been calculated erroneously.

Adulteration.—Native producers frequently adulterate gingergrass oil with oils of turpentine or coconut, or with kerosene. These adulterants can be detected by the methods described under "Indian Palmarosa Oil."

Chemical Composition.—The composition of the oil derived from *Cymbopogon martini* Stapf var. *sofia* has been investigated by the chemists of Schimmel & Co.,[3] among them chiefly Walbaum and Hüthig,[4] and Gildemeister and Stephan.[5] The following compounds, listed approximately in order of their boiling points, have been identified:

[2] "Die Ätherischen Öle," 3d Ed., Vol. II, 300.
[3] *Ber. Schimmel & Co.*, April (1904), 52; October (1904), 41; April (1905), 34; October (1906), 33. Cf. Gildemeister and Hoffmann, "Die Ätherischen Öle," 3d Ed., Vol. I, 544.
[4] *J. prakt. Chem.* [2], **71** (1905), 459.
[5] *Arch. Pharm.* **234** (1896), 326.

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d-Limonene. Preparation of its α-nitrolpiperidine m. 93°, and α-nitrolbenzylamine m. 93°.

Dipentene. Tetrabromide m. 125°; α-nitrolpiperidine m. 153°.

d-α-Phellandrene. Nitrite m. 120°.

An Aldehyde(?) $C_{10}H_{16}O$. The oil contains small quantities (about 0.2 per cent) of an aldehyde $C_{10}H_{16}O$, b_{754} 221°–224°, b_5 76°–78°, d_{15} 0.9351, α_D ±0°, n_D^{20} 1.47348, whose odor resembles that of citronellal and heptaldehyde. On reduction, the aldehyde yielded an alcohol $C_{10}H_{18}O$, the phenylurethane of which melted at 100°–101°. Oxidation by air gave an acid $C_{10}H_{16}O_2$, m. 106°–107°, which was observed also in the saponification lyes of the oil. The aldehyde was characterized, but not identified, by these derivatives: oxime m. 115°–116°, semicarbazone m. 169°–170°, semioxamazone m. 244°–245°, phenylhydrazone m. 63°, β-naphthocinchoninic acid m. 261°.

Dihydrocuminyl Alcohol (Perillyl Alcohol). This alcohol has an odor reminiscent of linaloöl and terpineol and yields a naphthylurethane m. 146°–147°. Its identity with perillyl alcohol was established by Semmler and Zaar.[6]

dl-Carvone. Characterized by the preparation of its semicarbazone m. 153°–154°.

Geraniol. Oil of gingergrass contains from 36 to 65 per cent of total alcohols, calculated as geraniol. The alcohols consist chiefly of geraniol, which was identified through its diphenylurethane m. 82°, and by oxidation to citral.

Suggested Additional Literature

F. Bruno, "Acclimatization and Commercial Utilization of *Cymbopogon flexuosus* Stapf, and of *Cymbopogon martini* Stapf var. *motia*." *Boll. studi inform. R. Giardino Col. Palermo* **13** (1934). *Parfums France* **13** (1935), 33. *Chem. Abstracts* **29** (1935), 3464.

OIL OF LEMONGRASS

Essence de Lemongrass *Aceite Esencial Lemongrass* *Lemongrasöl*
Oleum Andropogonis Citrati

Introduction.—Oil of lemongrass is one of the most important essential oils. Large quantities are used for the extraction of citral, the chief constituent of the oil. Citral is the starting material for the preparation of the important ionones (a series of aromatics with a powerful violet odor).

[6] *Ber.* **44** (1911), 460.

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Because of its high citral content (75–85 per cent) the oil possesses a strong lemon odor, whence the name lemongrass.

The plant grows wild in many tropical and semitropical parts of Asia, Africa, and America. For the extraction of its oil, however, only cultivated lemongrass is now employed, the collection of wild growing plants being too costly.

The trade distinguishes between two principal types of lemongrass oil, viz., the East Indian and the so-called West Indian oil. Both contain from 75 to 85 per cent of aldehydes (chiefly citral),[1] but the oils differ slightly in that the West Indian product is usually less soluble in 70 per cent alcohol than the East Indian. The lower solubility of the West Indian oil, particularly noticeable after storage of the freshly distilled oil, is due to the presence, in the foreruns, of myrcene, an olefinic terpene, which on exposure to air and light readily polymerizes. (Cf. "Physicochemical Properties" and "Chemical Composition.")

The East Indian oil is produced only in a small section of the southwestern part of India, near the Malabar Coast. It is the older of the two types of oil, large quantities having been exported annually for many years. The Indian industry, cultivation as well as distillation, is utterly primitive, and entirely in the hands of natives. Nevertheless, the East Indian oil has always been noted for its good quality, and particularly its high citral content. The high quality of the oil may be attributable to soil conditions, and perhaps also to altitude and climate, because the methods of cultivation and distillation employed in India cannot even be compared with those used for the production of the West Indian type of oil. More probably, however, the good quality of the Indian oil is to be attributed to the plant species from which it derives.

The term "West Indian lemongrass oil" is actually a misnomer. It came into use years ago when small quantities of oil were produced in the West Indian islands, largely on an experimental basis, and were found to differ from the East Indian oil, chiefly in possessing a lower solubility (see above). In order to differentiate this type of oil from the more soluble East Indian oil, the name "West Indian oil" was introduced, and has remained in use, although the oil is now produced in large quantities in several parts of the world: in Guatemala (Central America), Haiti (West Indies), São Paulo (Brazil), the Comoro Islands, Madagascar, and Indo-China. More or less successful experiments in cultivating the plant and distilling the oil have been undertaken in Puerto Rico and Dominica (West Indies), Honduras,

[1] The aldehydes present in lemongrass oil [illegible]

Mexico, Paraguay, and even in subtropical countries such as Florida (United States), success or failure depending upon conditions of climate, soil and altitude, and last but not least upon the cost of labor.

For some years prior to World War II Madagascar and the Comoro Islands produced from 50 to 60 metric tons, annually, of lemongrass oil, most of which went to France. A few years before the outbreak of the war Guatemala appeared on the market as a producer, shipping substantial quantities of oil to the United States, but fighting a gradually losing battle against competition from India. Madagascar, the Comoro Islands, French Indo-China, and Guatemala were then the only countries producing the West Indian type of lemongrass oil on a commercial scale. Fortunately for the Western Hemisphere, the Puerto Rico Experiment Station of the United States Department of Agriculture in Mayaguez, Puerto Rico, had been carrying on extensive experiments on the cultivation and distillation of lemongrass. The planting material raised at the Station became of great help to prospective producers in several parts of the Western Hemisphere, when the outbreak of World War II and transport difficulties reduced shipments of the East Indian oil. The ensuing sharp rise in price induced a number of planters in Guatemala, Haiti, and Brazil to grow lemongrass on a large scale, and production of the West Indian type of oil increased rapidly. The grass was grown on extensive plantations and distillation was carried out in efficient, modern equipment. The following figures will convey an idea of the extent of the American lemongrass oil industry by the end of the war: Guatemala produced annually about 120,000 lb., Haiti 50,000 lb., and Brazil 10,000 lb. of oil. The boom lasted until 1948, when the peak price of about $5.00 per pound of oil dropped to less than $1.00. This obviously discouraged many growers, who, on the basis of such low prices, could hardly cover their expenses, the cost of labor having risen sharply during the war. At the time of this writing (1949) the outlook is uncertain. The future of the American lemongrass oil will depend in large part upon competition offered by the East Indian oil. In the author's opinion the American lemongrass industry will be able to withstand competition from India only if it can mechanize part of its operations, particularly the harvesting of the grass, which in over-all calculations involves the greatest item of cost (labor). Very recently, considerable progress has been made in this direction, particularly in Guatemala.

The quality of the West Indian type lemongrass oil was once considered to be inferior to that of the East Indian oil, but the American oil i

when properly prepared, the American oil is beyond reproach. It can easily replace the Indian oil for the extraction of citral. The fact that the American oil, particularly after storage, exhibits a slightly lower solubility in 70 per cent alcohol than the East Indian oil, does not prevent its use for the scenting of soaps or technical preparations.

Botany.—There was much confusion, years ago, about the taxonomy of the plants which yield the East Indian and the West Indian types of lemongrass oil; however, Stapf [2] ended the long controversy by identifying the plant yielding the East Indian type of oil as *Cymbopogon flexuosus* (D.C.) Stapf, and the plant yielding the West Indian type of oil as *Cymbopogon citratus* (D.C.) Stapf. The two plants have been named variously also *Andropogon nardus* var. *flexuosus* Hack. or *A. flexuosus* Nees *ex* Steud., and *Andropogon nardus* var. *ceriferus* Hack. or *A. citratus* D.C., respectively.

The correctness of Stapf's classification was confirmed experimentally by Jowitt and Pickles,[3] who planted the two species in Ceylon, extracted the oils by distillation, and found that the oil derived from *Cymbopogon flexuosus* Stapf was soluble in 2.2 volumes, and more, of 70 per cent alcohol, whereas the oil derived from *C. citratus* Stapf was not clearly soluble even in 10 volumes of 90 per cent alcohol.

Experiments carried out at the Government Station in Barbados [4] (B. W. I.) with East Indian lemongrass seed imported from the State of Cochin (Malabar Coast) indicated that grass raised from this seed yielded an oil which was readily soluble in alcohol and contained a high percentage of citral.

Hood [5] does not contest the claim of Stapf that there are two distinct species of lemongrass, viz., *C. flexuosus* and *C. citratus*, but assumes the existence of numerous local varieties of *C. citratus*. The Bureau of Plant Industry has, in fact, experimented in the United States with thirteen varieties from eight different localities.

Lemongrass, a stoloniferous plant, is propagated by division of clumps; it does not ordinarily produce seed unless grown in a suitable climate without being cut for several years. For propagation the leaves of a fully matured healthy plant are cut back to about 3 in. One old clump yields as many as 50 new stools. The "bud pieces" or "seed pieces" are then planted in holes 2 to 3 ft. apart. At the Puerto Rico Experiment Station [6] it has been the practice to plant 4 bud pieces to the hill at intervals of 18 in. in rows 32 in. apart.

[2] *Kew Bull.* (1906), 297.
[3] Cf. *Circ. Agr. J. Roy. Botan. Gardens, Ceylon* **5**, No. 12 (1910), 137. *Bull. Imp. Inst.* **8** (1910), 144.
[4] Cf. *Ber. Schimmel & Co.*, October (1908), 76.
[5] *U. S. Dept. Agr., Bur. Plant Ind. Bull.* No. 442. *Am. J. Pharm.* **89** (1917), 180.
[6] Jones and Arrillaga, "Experiments with Lemongrass in Puerto Rico," *U. S. Dept. Agr., Fed. Expt. Sta., Mayaguez, Puerto Rico* (Publication forthcoming).

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Oil of Lemongrass East Indian

Producing Regions.—The East Indian lemongrass oil is produced in Travancore State, in the extreme southwestern part of India. The principal producing regions are confined to a relatively small section of this state and are located for the most part south of the Periyar River which empties into the Indian Ocean a short distance north of Cochin, the old seaport on the famed Malabar Coast. Starting from Cochin and traveling inland, due west, the visitor can reach the lemongrass regions in a day's drive, provided the conditions of the roads permit it. Most of the plantations are located in the foothills of the Ghats, a range of high and wild mountains stretching inland from north to south and paralleling the Malabar Coast.

Local conditions of soil, altitude, and climate in these hills and mountains vary so widely that it is very difficult to classify certain sections according to the quality of the oil and importance of production. In general, the northern sections of the producing regions, from which the bulk of the oil originates, produce a better quality of oil than the southern districts. Main producing centers in this area are the villages of Thoduhpuzha, Vazhakulam, Moovattupuzha, Perambavur, and Alwaye, the latter being an important trading center for the oil. These districts account for about 50 per cent of the total output of oil in southern India. The environs of Kothamangalom (also located in the north) supply about 40 per cent of the total output of Indian lemongrass oil.

Plant Varieties.—There are two types of lemongrass in Travancore State, differing in appearance and readily distinguishable by the color of the stems:

1. The so-called "red grass," locally known as "choomana poolu." The color of the stems is reddish. The leaves of this plant, the true *Cymbopogon flexuosus* Stapf, yield the normal East Indian lemongrass oil, containing 75 per cent or more of aldehydes (chiefly citral) and exhibiting good solubility. The bulk of Indian lemongrass oil is produced from the "red grass."

2. The so-called "white grass," locally known as "wella poolu." The color of the stems is whitish. It has been identified as *Cymbopogon flexuosus* Stapf var. *albescens*.[7] The oil derived from this plant possesses a low aldehyde content and poor solubility; moreover, a planting lasts only a few years. Only small quantities of this type of lemongrass are produced and that chiefly in the southern sections. According to Varier,[8] the oil from the white variety (which could not be identified botanically) contains camphene and dipentene, but no citral. The compound isolated from a sodium bisulfite

The oil derived from "white grass" cannot be marketed as normal lemongrass. Indian field brokers and intermediaries, therefore (to the annoyance of the exporters in Cochin), occasionally bulk small quantities of this oil with lots of normal oil, thus lowering the high aldehyde content and normal solubility of the latter. Perhaps the "white grass" is closely related to, if not identical with, the "Inchi grass" discussed below (p. 133).

Soil, Climate, and Altitude.—Lemongrass requires a warm tropical climate, plenty of sunshine, and intermittent, but not excessive, rainfall. It does not thrive in compact soils, which may retain pools of stagnant water. Warmth and sunshine conduce to the development of oil in the plant. In regions of abundant rainfall the plant may be harvested more frequently, during the year, than is possible in dryer regions, but the oil will be of lower citral content. On the higher slopes of the mountains, against which the monsoon clouds discharge their moisture, the grass contains a great deal of water and yields less oil than grass grown on the lower slopes, which are less exposed to the showers.

The quality of the soil exerts a considerable influence upon the yield of grass and oil per acre and upon the quality of the oil. The grass grows best on well-drained sandy loam; it thrives even on light sandy soils, provided they are sufficiently fertile. Plants from such sandy soil yield relatively more oil, and oil of higher citral content, than plants from very fertile soil.

Experimenting with the West Indian type of lemongrass (*Cymbopogon citratus* Stapf) in Florida, Hood [9] found that the moisture content of the soil also influences the citral content of the oil. Dry sandy soil yielded an oil containing 75 per cent of citral, a slightly humid sandy loam an oil containing 68 per cent of citral, and a humid loam an oil containing only 66 per cent of citral.

In southern India the growers obviously cannot use fertile soil for the raising of lemongrass, since such land is expensive and is required for other more vital crops—rice, tapioca, and cashew nuts, for example (the most important food crops). Most of the lemongrass plantings are therefore located on low-priced land on the slopes of hills, up to 2,000 ft. altitude.

Planting and Cultivating.—Until about forty years ago it was chiefly the wild growing grass which served for distillation of Indian lemongrass oil, because at that time buyers abroad did not insist upon a high citral content. Conditions have changed, however. Today it would be much too costly to collect wild growing grass, which is usually scattered over wide areas, and to separate it from other admixed grasses, the presence of which in the distillation material would yield oils of substandard citral content and poor solubility. For these reasons only cultivated lemongrass is at present used for distillation.

[9] *U. S. Dept. Agr., Bur. Plant Ind. Bull.* No. 442. *Am. J. Pha*

The plantations are scattered widely, and range in size from patch plantings, on jungle clearings, to fields of a hundred or even two hundred acres. The whole industry—cultivation of the grass as well as distillation—is entirely in the hands of natives, utterly primitive, and, as a village industry, forms part of the native's general agricultural activities. There is not a single plantation or distillery owned by white producers. Some of the patch fields and small plantings belong to individual villagers, but most of the land is owned by wealthy Indians, who cultivate it on their own account, with the help of hired labor, but only during periods of attractive prices. Whenever prices are low, they simply rent their land to small peasants on a sharecrop basis, or for very small fees—just enough to meet the land tax. The whole economic setup of the industry is such that, in years of very low prices, production diminishes, fewer peasants then being interested in operating the stills. Part of the equipment is left idle until distillation again becomes profitable.

Prior to planting, which in Travancore is by seed, the ground must be prepared, which is usually done toward the end of the dry, hot season (March or April). With the advent of the first, sporadic monsoon showers, the seed is sown by hand. Toward the end of April the showers become more frequent, and the plant develops rapidly. At this point, however, the grass is still too young and too short (being only about 1 ft. tall) to be harvested, as the yield of oil and the citral content would be substandard. While young, the grass must be protected by fences or guards against cattle or goats that may invade the field. The first harvest takes place near the end of May or the beginning of June, when the heavy monsoon rains begin. From then on, the leaves may be cut every 6 to 7 weeks; they grow up rapidly after each cutting.

The time of cutting exerts a considerable influence upon the yield and quality of the oil. Young and tender grass, harvested in the early season (during April, for instance), yields an oil of abnormally low aldehyde content (60 to 70 per cent) and of poor solubility. Later the aldehyde content of the oil increases to 75 per cent and more. A similar increase takes place as regards yield of oil. In general, the yield of oil is lower in the months of heavy rain (June to August) than in the dry months.

In December the monsoon rain ceases, and the dry, hot season begins; it lasts to March or April. During this period the plant develops an inflorescence and the leaves and stems shrivel up; but the root remains alive. With the arrival of the new monsoon rain in March or April the plant develops new life; it flourishes and the cutting can be started anew. Prior to the arrival of the new monsoon showers, the dry grass is usu[illegible]

upon the later growth of the plant and the yield of oil. Under these circumstances the first cutting in the new monsoon season may be normal, but the second and following cuttings are likely to give an abnormally low yield of grass and oil.

Throughout its life span a plantation should be weeded out, but when prices are low this practice is often neglected, with the result that the planting soon becomes overgrown with weeds.

Cutting is usually done with sickles. Harvesters tie the cut grass into bundles weighing ½ to ¾ lb. These are then gathered into larger bundles, which the harvesters carry on their heads to nearby field stills.

Depending upon soil and climatic conditions, a plantation lasts, on the average, from six to eight years. The yield of oil is small during the first year. It increases with the second, and reaches a maximum in the third and fourth years, after which it declines progressively. If the prices are low, a plantation may be discontinued prematurely, to make room for more lucrative produce—such as rice, tapioca, or ginger. On the average one acre yields 30 to 40 lb. of oil per year with four cuttings. Higher yields may be obtained on fields that are well managed.

Distillation.—Because of the very primitive nature of the Indian lemongrass industry, with its widely scattered plantings in the hills, lack of roads, and difficulties of transportation, there are no centrally located, large-scale distilleries.

The stills, as was noted above, are utterly primitive, made of copper, and not equipped with perforated grids. A wide, protruding palm thatched roof protects the still, the stacked up plant material, and the workers, from exposure to sun and rain. The average retort is 6 ft. high, 3 ft. wide, and has a capacity of about 200 gal. At the side, about 3 ft. above the bottom is a manhole through which the plant material can be charged and discharged. During distillation the manhole is closed with a cover, held fast by means of a mud paste which dries and hardens as the metal walls become hot. The retort, up to the manhole, is imbedded into a hearth made of mud and bricks. It houses the fire beneath the bottom of the retort. About 300 to 500 bundles of freshly cut grass material (weighing approximately 200 to 300 lb.) are packed tightly into the still, up to ¾ of its height. Forty to fifty gallons of fresh water are poured into the still, filling it up to ¼ of its height. Operation in these stills therefore follows the principle of water distillation, a good part of the plant material being immersed in boiling water (cf. Vol. I of this work, pp. 112, 120, and 142). Since most still tops are not removable, all the charging and discharging has to be done through the manhole, a lengthy and tedious task.

The condenser consists of a coil inserted into a large wooden barrel, about

with cold water drawn from a brook or spring, or, if this is not available, with water drawn by means of a bucket and a high lever from a specially dug well.

A wide, low tin beaker serves as a sort of Florentine flask (oil separator), the accumulating oil being carefully skimmed off with a spoon and poured into bottles. The distillation water is discarded and not returned into the still for use with the next batch of plant material.

Courtesy of Fritzsche Brothers, Inc., New York.

FIG. 3. Lemongrass oil production in Cochin, East India. A typical distillation post in the interior.

Wood, either cut from nearby jungles by the distiller's family or purchased elsewhere, serves as fuel for the fire beneath the still. Since wood is abundantly available in the jungles, the distillers do not use the exhausted grass as fuel. Moreover, the high atmospheric humidity and frequent rain showers prevent drying of the spent leaf material.

Distillation of one charge lasts about 3 hr., yielding ½ to ¾ of a bottle (= ¾ to 1 lb.) of oil. This means approximately 0.2 to 0.3 per cent of oil, calculated upon a charge of 300 lb. of fresh grass material. Exact data are not available, because the plant charge is never weighed, and the yield of oil fluctuates greatly with the season, the condition of the plant material, its moisture content, and the age of the planting.

The distillers and their families work 15 hr. or more a day in the distillery and, by completing five charges, are able to produce 2½ to 3½ bottles of oil per day.

We should not close the section on the distillation of lemongrass without

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once more emphasizing the fact that in India, the oil is produced by distillation of leaf material while it is partly immersed in boiling water. The West Indian type of lemongrass oil, on the other hand, is obtained by steam distillation with steam supplied from a separate steam generator. In literature the opinion has occasionally been advanced that much of the insolubility of steam distilled lemongrass oil results from the fact that the higher boiling and less soluble fractions are carried over during the process of direct steam distillation, and that many of these would be left behind if the grass were immersed in water during distillation. Experiments undertaken at the Puerto Rico Experiment Station in Mayaguez, United States Department of Agriculture,[10] with a view to comparing yield, solubility, specific gravity, and citral content of the oils obtained by passage of steam through the grass, and by distillation of immersed grass indicate that material immersed in boiling water gave a slightly higher yield of oil (0.385 per cent against 0.375 per cent, calculated upon dried grass), a notably higher specific gravity (0.8945 against 0.8774), and a slightly higher citral content (81.66 per cent against 80.27 per cent) than the leaves distilled with steam. This is rather surprising, as purely theoretical considerations would lead to the conclusion that during distillation with boiling water part of the citral would polymerize.

Courtesy of Fritzsche Brothers, Inc., New York.

FIG. 4. Lemongrass oil production in Cochin, East India. A native distiller scooping the oil from the receiver into a bottle.

In both cases the oils were soluble in 3 volumes of 70 per cent alcohol. These experiments, however, cannot be accepted as fully conclusive, and further study will be required. From the practical point of view, however, the problem is not important, as large-scale distillation of plant material immersed in boiling water would be uneconomical. It is only in the small field stills employed in India, where labor is cheap, that distillation with boiling water can be effected.

[10] *Rept. Puerto Rico Exp. Sta.* (1939), 34.

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As regards drying of the plant material, the Indian producers, as we have seen, use freshly cut leaves for distillation.

Experimenting with the West Indian type of lemongrass in Florida, Hood [11] found that drying of the grass prior to distillation results in only small losses of oil. The yields of oil in his experiments were: 0.37 per cent from fresh grass, 0.31 per cent from sun-dried grass, and 0.32 per cent from artificially dried grass, all yields calculated on the basis of fresh grass. The citral content in the three experiments (determined by the sulfite method) was 77 per cent, 78 per cent, and 79 per cent, respectively. The citral content was thus about equal in the three samples. The solubility of the oils, however, was affected by drying of the leaves: the solubility of oils distilled from dried grass decreased more rapidly on aging than the solubility of oils distilled from fresh material.

Similar experiments carried out by the Puerto Rico Experiment Station, United States Department of Agriculture,[12] led to conclusions significant in field management: it was found that when grass is dried to 45 to 66 per cent of its original weight, it nevertheless yields almost the same amount of oil and content of citral as fresh grass; but the drying results in a great saving of field labor. The procedure would also effect an economy in the fuel required for distillation.

Period of Drying in Sunlight	*Moisture Content of Grass in Per Cent*	*Yield of Oil, Calculated on Weight of Fresh Grass, in Per Cent*	*Specific Gravity of Oil*	*Citral Content of Oil in Per Cent*
Distilled Immediately...	80.27	0.348	0.8945	77.50
One Day's Drying.....	79.69	0.345	0.8900	76.60
Two Days' Drying.....	77.82	0.287	0.8924	73.00
Three Days' Drying....	71.22	0.244	0.9001	73.04
Four Days' Drying.....	66.50	0.344	0.8970	77.84
Five Days' Drying.....	43.50	0.336	0.8954	79.00

As was to be expected, exposure to sunlight progressively reduced the yield of oil for the first three days on the basis of both the fresh and the treated leaves. However, on the fourth day there was a surprising increase in yield of oil, which was still further increased on the basis of the treated grass on the fifth day.

Handling and Marketing of the Oil.—As has already been mentioned, the distillers in India collect the crude oil in bottles. At the time of collection it still contains small quantities of water and copper salts and has a turbid appearance. Like so much of the native produce of the Orient, the oil has

[11] *U. S. Dept. Agr., Bur. Plant Ind. Bull.* No. 442. *Am. J. Pharm.* **89** (1917), 180.
[12] *Rept. Puerto Rico Exp. Sta.* (1939), 32.

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to pass through the hands of several field brokers, intermediaries, and dealers (all natives), before it reaches the "Go-downs" (warehouses) of the exporters in the port of Cochin.

The distiller is often "financed" by a field broker, whose "capital" consists of a few hundred rupees, and who regularly visits the distillation posts to collect the oil in cans and carry it back to the village where he has his "office." Or the distiller himself brings the oil to the nearest village and sells it to any of the numerous small shops which purchase lemongrass oil and other agricultural crops, often on a sort of barter basis. The field brokers and shop owners then filter the oil through paper into tin cans (old gasoline cans). These men, in turn, are financed by larger dealers, most of them located in the village of Alwaye, the principal trading center of lemongrass oil in the producing regions. Some of these brokers and merchants are relatively wealthy and act as purchasing agents for exporters in Cochin, enjoying their confidence and buying all kinds of native produce for them (lemongrass oil, ginger, etc.). Whenever the exporters in Cochin receive shipping orders from abroad, they either draw from stocks in their own warehouses or call upon their purchasing agents in the larger villages for additional supplies.

Every exporter has a simple "laboratory," where incoming cans of oil are tested for solubility in 70 per cent alcohol and for citral content (by the bisulfite method). Poor oils are rejected, whereas oils on the borderline are bulked with larger lots of good quality. Standard lots of oil, containing at least 75 per cent of citral, are thus made up and shipped abroad in galvanized iron drums.

From experience the exporters—many of them branches of European import and export houses—know that on aging lemongrass oil gradually loses some of its citral content, the loss being about ½ per cent per month. Since the shipping contracts are entered on the basis of a minimum content of 75 per cent of citral (bisulfite method), the exporters standardize their lots in Cochin to a minimum of 77 per cent of citral, which would allow for the loss of some citral during the transport abroad.

Thus each lot of oil shipped abroad consists of bulkings of many small lots. For this reason, oils of high citral content are locally much in demand and at a premium; they are required to counterbalance lots of lower citral content.

In times of high prices, the native dealers in the villages occasionally adulterate their oil lots by the addition of fatty oils or kerosene; but such additions lower the citral content of the oil and its solubility, and the exporters in Cochin will probably reject such lots. A form of adulteration more difficult to detect by t[redacted]

sists in the addition of methylated spirits. In times of low prices adulteration of lemongrass oil hardly pays, and is not frequently encountered.

The best time for purchasing lemongrass oil in Cochin, for shipment, is at the end of the main producing season (November to January). Great quantities of oil are then available, usually at low prices, unless the oil is held by some of the wealthy Indian dealers for the purpose of speculation. Immediately after the producing season, the oil has the highest content of citral, which is another reason why the oil should be shipped at this period. Buyers abroad should insist upon oils of most recent harvest.

Exports of Oil.—In the five years preceding World War II, India exported the following quantities of lemongrass oil:

	Hundredweight
1934	6,062
1935	7,067
1936	6,956
1937	7,146
1938	7,694

During and right after the war the figures fluctuated considerably, as can be seen from these statistics: [13]

Fiscal Year (Ending March 31st)	*Quantity of Oil in Gallons*
1939–1940	100,135
1940–1941	83,535
1941–1942	122,924
1942–1943	59,452
1943–1944	54,306
1944–1945	121,629
1945–1946	150,790

According to Volkart Brothers, Inc.,[14] New York, the exports of East Indian lemongrass oil (in long tons) for the last few years have been as follows:

1942–1943	114
1943–1944	382
1944–1945	566
1945–1946	505
1946–1947	465
1947–1948	232

[13] "Accounts Relating to the Sea-borne Trade and Navigation of British India," *India Commercial Intelligence Statistics Dept.*, March (1946).

[14] Courtesy Mr. E. H. Sennhauser, Secretary.

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During the 1947–1948 season, the United States took 85 tons or 38.33 per cent, and the United Kingdom 52 tons or 23.64 per cent of the total exports.

Physiochemical Properties.—Oil of lemongrass is a mobile, yellow to reddish-brown oil with a powerful lemon-like odor.

The quality of the oil is determined by its content of aldehydes (chiefly citral, and usually expressed as citral), which varies from 70 to 85 per cent (assayed by the bisulfite method). Commercial contracts are entered on the basis of a minimum citral content of 75 per cent (bisulfite method). When using the neutral sulfite method, the values, according to Schimmel & Co.,[15] will be 2 to 5.5 per cent lower. This difference may be explained by the fact that the bisulfite method determines not only the citral but also the small quantities of other aldehydes present in the oil, and a part of the methyl heptenone. Both, the bisulfite and the neutral sulfite method, give the percentage of citral by volume, whereas the hydroxylamine method gives it by weight. (For details the reader is referred to Vol. I of this work, pp. 279, 283, 285.)

The British Aromatic Compound Manufacturers' Association [16] proposed replacing the bisulfite method, used for many years for the assay of lemongrass oil, with the more exact hydroxylamine hydrochloride method.

Analysis of 24 samples of lemongrass oil by the hydroxylamine hydrochloride method showed 74 to 75.8 per cent (*by weight*), an average of 74.84 per cent of citral, whereas examination of 17 samples by the bisulfite method showed 73.5 to 81 per cent (*by volume*), an average of 75.66 per cent of citral.

Determinations made by members of the Sub-Committee lead to the conclusion that the maximum variation with the hydroxylamine hydrochloride method should not exceed ± 1 per cent.

The hydroxylamine hydrochloride method is undoubtedly the more exact assay, especially in the hands of a skilled analyst; but in the shipping ports of India and other producing countries the necessary laboratory equipment and skill may not always be available.

The export contracts of lemongrass oil should always specify the method by which the citral content of the oil has been assayed.

As has been pointed out above, the content of citral decreases gradually on storage and aging of the oil. This decrease is usually accompanied by an increase in the specific gravity of the oil.[17] For example, an oil with a

[15] *Ber. Schimmel & Co.*, October (1908), 76.

[16] *Perfumery Essential Oil Record* **39** (1948), 255.

[17] Cf. *Ber. Schimmel & Co.*, October (1909), 65; April (1913), 67. Umney and Bunker, *Perfumery Essential Oil Record* **4** (1913), 38. Parry, *ibid.*

citral content of 78 per cent and a specific gravity of 0.889, after two years of storage, exhibited a citral content of only 60 per cent and a specific gravity of 0.895. In a sample, which contained moisture, the citral content decreased from 79 to 68 per cent within three months, whereas another sample, which had been carefully dried, under the same conditions, exhibited a decrease, in citral content, from 79 to 74 per cent only.

It is therefore important, in the storage of lemongrass oil, to exclude air and light, and to dry the oil before it is filled into the storage tanks. Incidentally the oil should not be handled (filtered, etc.) too often in open air, as this may affect the citral content.

Experimenting with the West Indian type of lemongrass oil, the Puerto Rico Experiment Station, United States Department of Agriculture,[18] found that samples of lemongrass oil exposed to air *and* light suffered the most physical and chemical changes. Their solubility was lowered considerably, the citral content decreased (from 86.07 to 78.81 per cent, after eleven months), and the specific gravity increased (from 0.8971 to 0.9055, after eleven months). The freshly distilled oils were soluble in 3 volumes of 70 per cent alcohol; after eleven months they were soluble in 3.7 volumes of 80 per cent alcohol. At the end of the experiments (eleven months) the oil was viscous and resinous.

Other oil samples exposed to air, but not light, did not deteriorate so much, at least as regards citral content and solubility, which would indicate that light is a detrimental factor. Curiously, the specific gravity of these samples, on standing, increased to a much greater extent (from 0.8719 to 0.9364, after eleven months) than the samples exposed to periodic daylight (see above).

Samples protected from both air and sunlight changed little, their properties remaining practically stable.

Gildemeister and Hoffmann[19] reported the following properties of East Indian lemongrass oil:

Specific Gravity at 15°	0.899 to 0.905, exceptionally as low as 0.895 and as high as 0.911 in otherwise good oils
Optical Rotation	$+1° 25'$ to $-5° 0'$
Refractive Index at 20°	1.483 to 1.488
Aldehyde Content:	
Bisulfite Method	70 to 85%
Neutral Sulfite Method	65 to 80%
Solubility	Soluble in 1.5 to 3 vol. of 70% alcohol; occasionally with opalescence or turbidity, due to separation of paraffins

[18] *Rept. Puerto Rico Exp. Sta.* (1939), 35.
[19] "Die Ätherischen Öle," 3d Ed., Vol. II, 310.

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Shipments of genuine East Indian lemongrass oil received and analyzed by Fritzsche Brothers, Inc., New York, had properties varying within these limits:

Specific Gravity at 15°/15°	0.899 to 0.911; usually 0.900 to 0.906
Optical Rotation	−1° 10′ to −3° 10′
Refractive Index at 20°	1.4855 to 1.4899
Aldehyde Content (Bisulfite Method)	71.8 to 79.1%
Solubility at 20°	Usually soluble in 2 to 2.5 vol. of 70% alcohol; occasionally opalescent or slightly cloudy with more. A few lots were not clearly soluble in 70% alcohol, up to 10 vol.

Chemical Composition.—The main constituent of lemongrass oil was first identified by Bertram [20] and named citral because of its strong lemon odor. The configuration of this aliphatic terpene aldehyde has been the subject of much controversy. For details the reader is referred to the monograph on "Citral" in Volume II of this work, p. 326.

The following substances (listed approximately in order of their boiling points) have been identified in the East Indian type of lemongrass oil derived from *Cymbopogon flexuosus* Stapf (cf. "Chemical Composition of Oil of Lemongrass West Indian").

Myrcene(?). Literature does not offer much evidence that this aliphatic terpene actually occurs also in the East Indian type of lemongrass oil. It is quite an important constituent of the West Indian oil; in fact, the latter oil differs from the former mainly in its content of myrcene. In any case, the East Indian oil appears to contain much less myrcene than the West Indian oil does.

Methyl Heptenone. Identified by Barbier and Bouveault,[21] and by Schimmel & Co.[22]

Limonene(?) and Dipentene. The presence of dipentene and perhaps limonene in lemongrass oil has been reported by Stiehl.[23]

A Terpene(?). Barbier and Bouveault [24] noted still another terpene b. 175°, α_D −5° 48′ in some lots of lemongrass oil. It yielded a liquid bromide, which on standing separated small quantities of a solid substance m. 85°. The presence of this terpene in some oils perhaps can be explained by adulteration of the lots in question.

Methyl Heptenol. Isolated by Elze [25] who oxidized the alcohol (b_{759} 178°–180°, d_{15} 0.8579, α_D −2°) to methyl heptenone and prepared the semicarbazone m. 135°–136° of this ketone.

[20] *Ber. Schimmel & Co.*, October (1888), 17. Cf. Tiemann, *Ber.* **32** (1899), 115, 117; **33** (1900), 877.
[21] *Compt. rend.* **118** (1894), 983.
[22] *Ber. Schimmel & Co.*, October (1894), 32. Cf. Tiemann and Semmler, *Ber.* **28** (1895), 2126, footnote.
[23] *J. prakt. Chem.* [2], **58** (1898), 51; **59** (1899), 497.
[24] *Compt. rend.* **118** (1894), 983.
[25] *Riechstoff Ind.* **4** (1929), 23.

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Linaloöl(?). Schimmel & Co.[26] reported that linaloöl is probably present in the fraction b. 198°–200°.

Citronellal(?). Doebner [27] expressed the opinion that the oil contains citronellal. It is known that citronellal occurs in East Indian lemongrass oil in quantities of 1 per cent only; in some lots of oil this aldehyde seems to be absent.

n-Decylaldehyde. According to Schimmel & Co.,[28] oil of lemongrass contains small quantities of *n*-decanal, which yielded a semicarbazone m. 102° and could be oxidized to *n*-capric acid.

An Aldehyde(?) $C_{10}H_{16}O$. Schimmel & Co.[29] also noted traces of an aldehyde $C_{10}H_{16}O$, b_6 68°, d_{15} 0.9081, α_D +0° 50′, n_D^{20} 1.45641, which gave a semicarbazone m. 188°–189°, and which was isomeric with citral.

Citral *a* and *b*. The main constituent of East Indian lemongrass oil. Good quality lemongrass oil contains a minimum of 75 per cent of citral (bisulfite method).

Nerol. Elze [30] identified nerol by the preparation of its diphenylurethane m. 52°, and its tetrabromide m. 118°.

Geraniol. This terpene alcohol is contained, free and as ester, in the higher boiling fractions of the oil. Tiemann and Semmler [31] isolated geraniol through its calcium chloride compound and identified the regenerated alcohol by preparation of its diphenylurethane m. 82°.

Farnesol. Elze [32] characterized this sesquiterpene alcohol in the fraction b_5 140°–145° by oxidizing it to farnesal and by preparing the semicarbazone m. 134° of farnesal.

Undoubtedly, East Indian lemongrass oil contains quite a number of still other constituents which are present in small quantities and in traces. Therefore, they can be isolated and identified only when large quantities of the oil are processed.

Use.—Because of its powerful lemon-like odor, oil of lemongrass is used widely for the scenting of soaps, detergents, and all kinds of technical products. The bulk of the oil, however, is employed for the isolation of citral, which, as such, is used in flavors, cosmetics, and perfumes, or is converted into ionones, a group of very important synthetic aromatics possessing a strong and lasting violet odor.

As the starting material for the preparation of the ionones, oil of lemongrass has become one of the most important and indispensable essential oils in the course of the past fifty years. This applies to the East Indian,

[26] *Ber. Schimmel & Co.*, October (1894), 32; April (1899), 73.
[27] *Ber.* **31** (1898), 1891.
[28] *Ber. Schimmel & Co.*, October (1905), 42.
[29] *Ibid.*, 43.
[30] *Riechstoff Ind.* **4** (1929), 23.
[31] *Ber.* **26** (1893), 2721. Cf. Elze, *Riechstoff Ind.* **4** (1929), 23.
[32] *Riechstoff Ind.* **4** (1929), 23.

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as well as to the West Indian, type of lemongrass oil. Large quantities of β-ionone are now employed for the manufacture of synthetic vitamin A.

Oil of Lemongrass West Indian

As was pointed out in the introduction to the monograph on "Lemongrass Oil," the West Indian type of oil has attained great importance since the outbreak of World War II, when the East Indian oil was no longer readily available, due to shipping difficulties. Extended plantations of the so-called West Indian type of lemongrass, *Cymbopogon citratus* (D.C.) Stapf, were started in several parts of the Western Hemisphere, and large quantities of oil have since been produced. The quality of the oil is now excellent, except perhaps in respect to solubility in 70 per cent alcohol, a factor of little importance.

Lemongrass Oil from Madagascar and the Comoro Islands

The Madagascar and the Comoro Islands have been supplying lemongrass oil, West Indian type, regularly for quite some years. Even before World War II, 50 to 60 metric tons were produced annually on these East African islands and exported (chiefly to France). The oil has been of good quality.

The principal producing regions are located on Madagascar proper, in the most northern part, opposite the small island of Nossi-Bé. The Comoro Islands (located between Madagascar and Zanzibar) also supply substantial quantities of the oil.

The plant was introduced to Madagascar and the Comoro Islands long ago, probably from the French West Indies at the time that France started to colonize those parts of the world. The lemongrass does not seem to occur wild in Madagascar and on the Comoro Islands. Only cultivated plants are used for distillation. The plantations, owned chiefly by large-scale European (French) planters, are extensive, well kept, and play an important secondary role in the operation of agricultural enterprises on the island. Since natural pastures for the grazing of cattle are few in Madagascar and on the Comoros, the cattle have to be fed, in part, with lemongrass, after the oil has been removed by distillation, and after the residual plant material has been sweetened by the addition of a little molasses from sugar cane. Thus, the essential oil, which must first be extracted to make the grass palatable to cattle, becomes almost a by-product.

The grass requires much sunshine, occasional rainfall, a warm tropical climate, and some atmospheric humidity, even during the dry season. Excessive rainfall is detrimental to the quality of the oil, as during periods of

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heavy rains the citral content will be about 5 per cent lower than during the dry season. The grass flourishes best on a well-drained, sandy, or sandy-loamy soil.

Prior to planting, the soil must be loosened by thorough plowing. Planting usually takes place from December to January (the rainy season). The plants are reproduced by root division (segments), and not by seed, since *Cymbopogon citratus* very rarely develops flowers and seed. Holes about 10 in. deep are dug into the ground with a crowbar, and the root segments are planted 1.5 or 2 ft. apart, in rows about 3 ft. apart. The holes are tightly filled with earth to facilitate development of a good root system. Depending upon climatic and soil conditions, the young plants require from four to six months to develop sufficiently to be cut for the first time. After the first harvest, the grass grows rapidly, and, depending upon the rainfall, can be cut every three or four months. In other words, three or four annual harvests are possible. The cutting is done by hand (machetes), with the help of hired native labor. In the Comoro Islands the cut leaf material is sometimes left lying in the fields for three or four days before being transported to the distilleries. In Madagascar the leaves are usually distilled right after cutting. No strict rules hold, however, in this respect, all depending upon the daily exigencies.

A planting should last at least four years. If it is started on fertile ground, and properly cared for, its life span may be nine years. Lemongrass is quite a soil-exhausting plant, and requires potash and phosphate fertilizers. After lemongrass, leguminous plants such as peas should be planted to enrich the soil again. After six months, the leguminosae may be plowed under.

The grass is distilled either by direct steam distillation or by "steam *and* water" distillation. The direct steam stills have a capacity of about 3,000 liters, a charge consisting of about 800 to 1,000 kg. of plant material. The pressure in the steam generator is about 7 kg. Distillation of one charge lasts about 1 hr. During the height of the season up to fourteen charges are distilled daily in each still. The distilleries, located on Madagascar proper, and on Nossi-Bé, usually contain three to five stills each.

The "steam *and* water" stills also have a capacity of about 3,000 liters. The plant material is supported by a perforated grid, beneath which water is brought to a boil by a steam jacket. In the steam jacket the steam pressure is 3 to 3.5 kg. Distillation of one charge lasts for several hours, because the steam pressure is much lower than in direct steam stills.

Exhausted, sun-dried grass

One hectare of lemongrass yields from 50 to 80 kg. of oil per annum, the yield depending, of course, upon the age of the plantation. (This represents a yield of approximately 44 to 71 lb. of oil per acre and per year.)

In comparison with these figures, the Puerto Rico Experiment Station, U. S. Department of Agriculture,[33] obtained experimental yields of lemongrass oil ranging from 67.65 lb. to 98.18 lb. per cutting and per acre, four cuttings being made in one year. The total yield of oil per year and per acre in 1938 was 312.05 lb.

Courtesy of Fritzsche Brothers, Inc., New York.

FIG. 5. Production of lemongrass oil in Madagascar. View of a distillery on one of the plantations.

The quality of the oil produced in Madagascar and on the Comoro Islands is good, the physicochemical properties being normal for the West Indian type of lemongrass oil.

Chemical Composition.—The fact that citral is the main constituent of West Indian (as well as of East Indian) lemongrass oil has been known for a long time. Years ago, Umney and Bennett[34] observed, however, that on fractional distillation the two types of oil behave differently. The East Indian oil starts boiling only above 210°, whereas, at that temperature, 23 per cent of the West Indian oil has already distilled over. Comparative distillation tests at reduced pressure, and separation of each oil into five equal fractions, showed that the physicochemical properties of the corre-

[33] *Rept. Puerto Rico Exp. Sta.* (1939), 30.

[34] *Chemist Druggist* **70** (1907), 138. Cf. DeJong, *Teysmannia* (1907), No. 8. Watts and Tempany, *West Indian Bull.* **9** (1908), 265. *Ber. Schimmel & Co.,* April (1909), 58; October (1908), 75.

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sponding fractions of the two oils displayed considerable differences: the West Indian type yielded optically inactive fractions only, whereas the fractions of the East Indian oil had rotations ranging from $-12°$ to $-2°$. As far as the specific gravity is concerned, the first fraction of the West Indian oil (20 per cent of the total) assayed 0.821, whereas that of the East Indian type was 0.882. This indicates the presence of an olefinic terpene (myrcene!) in the West Indian oil. As a matter of fact, the two types of oil differ principally in the occurrence of substantial quantities of myrcene in the West Indian oil. The presence of myrcene in this oil also influences its solubility in dilute alcohol. Freshly distilled West Indian oils are soluble in 70 per cent alcohol but lose their solubility on aging, which fact is probably caused by diene-condensation and polymerization of myrcene.

According to Naves and Auriol,[35] both types of lemongrass oil contain from 75 to 85 per cent of citral, but the West Indian oil also contains some other aldehydes (aside from citral) which are difficult to remove from the citral and may cause trouble in the technical separation and purification of citral.

The most complete investigation of the West Indian type of lemongrass oil (origin: equatorial Africa and Comoro Islands), derived from *Cymbopogon citratus* Stapf, is that of Naves,[36] who noted the presence of the following compounds:

Isovaleraldehyde. In the fraction b. 90°–110°. Oxidation with silver oxide in an alkaline medium gave a valeric acid, the silver salt of which contained 51.31 per cent of silver.

Furfural. This content of silver indicated the presence (in much smaller quantity) of furfural. Confirmed by color reaction.

Myrcene. The largest constituent (next to citral) of the West Indian type of lemongrass oil is myrcene, which comprises 12–19 per cent of the oil produced in the Comoro Islands, and 18–20 per cent of the oil distilled in equatorial Africa. The isolated myrcene, rectified repeatedly over metallic sodium, had these properties: b. 168°–169°, b_{12} 53°–55°, d_{15} 0.7943, α_D ±0°, n_D^{15} 1.4698. Hydrogenation with sodium and alcohol gave dihydromyrcene which yielded a tetrabromide m. 93°–94°. (Incidentally the West Indian type of lemongrass oil is the best raw material for the isolation of myrcene.)

Dipentene. Aside from some slightly laevorotatory monocyclic terpenes, the terpene fraction of the oil, b_{12} 55°–58°, contained traces of dipentene which was identified through its tetrabromide m. 124°.

Methyl Heptenone. Isolated from the fraction b_{12} 55°–65°, by means of the solid bisulfite compound, and identified through its semicarbazone m. 136°–137°. The total oil contains 0.2–0.3 per cent of methyl heptenone.

[35] *XVII Congrès de Chimie Industrielle, Paris* (1937), 83.

An Aldehyde(?) $C_{10}H_{16}O$. On treating the fraction b_{12} 78°–88° with a 35 per cent solution of bisulfite, Naves obtained a mixture (yield 0.05 per cent of the total oil) rich in aldehydes, the most typical fraction of which had these properties: b_{10} 78°–80°, d_{15} 0.9068, n_D^{21} 1.4631; semicarbazone m. 191°–192°. This aldehyde exhibited a disagreeable, camphor-like odor. It is, perhaps, identical with the aldehyde $C_{10}H_{16}O$ which Schimmel & Co.[37] had noted in East Indian lemongrass oil, and which gave a semicarbazone m. 188°–189°.

Citronellal. Observed by Naves [38] in the fraction b_{12} 88°–93°. Separation of citronellal by means of the solid bisulfite compound was unsatisfactory and gave an impure product, probably contaminated with the aforementioned aldehyde $C_{10}H_{16}O$. By repeated fractionation accompanied by heavy losses, Naves obtained a substance b_{10} 88°–89°, d_{15} 0.859, α_D −0°23′, n_D^{15} 1.4504. The content of citronellal in West Indian lemongrass oil hardly exceeds 0.1 per cent.

n-Decylaldehyde. Separated from the fraction b_{12} 93°–100° by means of the solid bisulfite compound, and identified by preparation of the semicarbazone m. 101°–102°. The content of *n*-decylaldehyde in West Indian lemongrass oil is less than 0.1 per cent; in some lots of oil the aldehyde seems to be absent.

An Aldehyde or Ketone(?). From the mother liquors of extraction, Naves regenerated a mixture of substances (with an odor of acetophenone) n_D^{15} 1.5110, giving Tollens aldehyde reaction. A semicarbazone melted at 229°–231°. The oil examined contained only minute traces of this aldehyde or ketone.

Citral *a* and *b*. The chief constituent of West Indian lemongrass oil is present in the fraction b_{12} 104°–106° as a mixture of the *a* and *b* isomers. The respective semicarbazones melted at 162°–164°, and at about 132° (not clearly).

According to Naves,[39] the West Indian type of lemongrass oil contains 65–86 per cent of citral (determined by the sodium bisulfite method).

Farnesal. Using the bisulfite method, Naves isolated very small quantities of farnesal from the high boiling fractions b_5 130°–180° of the oil, 120 kg. of oil giving only 12 g. of farnesal $b_{2.5-3.0}$ 135°–140°, d_{17} 0.896, α_D ±0°, n_D^{17} 1.4988; semicarbazones m. 133°–134°, and m. 134°–135°.

α,β-Dihydropseudoionone. From the odor of the first fraction of farnesal, Naves suspected the presence of α,β-dihydropseudoionone, whose occurrence along with farnesal may be anticipated by reason of their genetic relations, which are analogous to those of methyl heptenone and citral. After destruction of the aldehydes by silver oxide, Naves obtained a semicarbazone from the neutral fraction which, on mixing with the semicarbazone m. 94° of α,β-dihydropseudoionone (obtained by splitting of the farnesal isolated from the oil), melted at 91°–92°.

Esters: (Iso?) Valerates, Caprylates, Caprates, Citronellates, Geranates, and Nerates. The esters present in the West Indian type of lemongrass oil are contained in the fractions distilling after citral. They consist chiefly of (iso?) valerates, caprylates, caprates, citronellates, and geranates.

Citronellic acid could not be obtained in pure form but was evidenced by the properties of the fraction b_{15} 115°–125°, d_{15} 0.9453, n_D^{20} 1.4610. The higher [illegible]

d_{15} 0.9730–0.9690, α_D ±0°, n_D^{20} 1.4836–1.4809, and undoubtedly was accompanied by its isomer, viz., neric acid. On reduction with sodium and alcohol, geranic acid gave (75 per cent yield) citronellic acid b_6 133°–135°, d_{15} 0.9291, α_D ±0°, n_D^{15} 1.4553, whereas on treatment with 75 per cent sulfuric acid at room temperature it cyclized to isogeranic acid m. 105°.

Alcohols. The alcohol fraction b_{12} 60°–98° of West Indian lemongrass oil is rich in tertiary alcohols, as evidenced by the difference in the percentage of free alcohols found by acetylation and by cold formylation (63.2 and 93.4 per cent, respectively).

Methyl Heptenol. Naves isolated methyl heptenol b. 176° (corr.), b_{12} 76°–78°, d_{15} 0.8581, α_D −1° 15′, n_D^{20} 1.4506 by means of its boric ester, and identified it through its allophanate m. 98°–99°.

l-Linaloöl. B_{10} 80°–82°, d_{15} 0.873, α_D −6° 30′, n_D^{20} 1.4620; identified through its phenylurethane m. 63°–64°.

α-Terpineol. B_{12} 96°–98°, d_{15} 0.9323, α_D −0° 36′, n_D^{20} 1.4884; identified through its phenylurethane m. 111°–112°.

Isopulegol. The impure α-terpineol was accompanied by some isopulegol which, on chromic oxidation, gave isopulegone, the semicarbazone of which melted at 172°.

The minimum content of these secondary and tertiary alcohols in the oils examined by Naves was 0.5 per cent, one-tenth of which was α-terpineol.

Geraniol. After the oil had been freed from aldehydes and saponified, the fraction b_{12} 98°–118° represented 0.5 to 0.9 per cent of the total oil, and contained 70 to 75 per cent of primary alcohols. From this fraction Naves isolated geraniol b_{12} 114°–115°, d_{15} 0.8833, α_D ±0°, n_D^{15} 1.4784 by means of its calcium chloride addition product, and identified it through its silver phthalate m. 132°–133°.

Nerol. The alcohols which did not react with calcium chloride contained some nerol which was characterized through its tetrabromide m. 117°–118°, and its diphenylurethane m. 52°.

Citronellol. Whether the oil contains citronellol remains an open question. The properties b_{12} 114°–116°, d_{15} 0.8813, α_D −0° 21′, n_D^{20} 1.4795 of the mixture (resulting from the phthalic treatment of the total fraction) exclude the possibility that an appreciable quantity of citronellol is present.

Farnesol. The fraction b_5 140°–146°, d_{15} 0.892, α_D −0° 12′, n_D^{15} 1.4897, on oxidation in the usual way, gave farnesal (semicarbazone m. 134°–135°) which indicates that the parent alcohol in question was farnesol.

A Bicyclic Sesquiterpene(?). The sesquiterpene fraction amounted to 1 per cent of the total oil. The sesquiterpenes consisted chiefly of a bicyclic sesquiterpene b_8 124°–126°, d_{15} 0.9166, α_D −9° 10′, n_D^{15} 1.5034, which was accompanied by monocyclic sesquiterpenes b_8 128°–132°, d_{15} 0.8984, α_D ±0°, n_D^{15} 1.4978. No crystalline chlorides or bromides could be obtained.

α-Camphorene and Other Diterpenes(?). The diterpene fraction of the oil investigated by Naves was comparatively large, amounting to 2–3 per cent of the total oil. It appeared to contain some optically inactive α-camphorene, along with

In the water oil (obtained by redistillation of the distillation waters) Naves observed *diacetyl, acetone, eugenol,* and other *phenols*(?).

To summarize, the West Indian type of lemongrass oil has the following composition:

Terpenes..... Myrcene (12–20%)
Dipentene (traces only)

Alcohols (1–1.5%)... Methyl Heptenol
Linaloöl
α-Terpineol
Geraniol
Nerol
Citronellol(?)
Isopulegol
Farnesol

These alcohols occur in the oil free or esterified with
Isovaleric Acid
Caprylic Acid
Capric Acid
Citronellic Acid(?)
Geranic Acid, and
Neric Acid(?)

Aldehydes.... Citral *a* and *b* (65–86% by the Bisulfite Method) and other aldehydes (less than 0.1%):
Furfural
Isovaleraldehyde
Decylaldehyde
Citronellal
An aldehyde $C_{10}H_{16}O$; semicarbazone m. 191°–192°
An aldehyde or ketone; semicarbazone m. 229°–231°
Farnesal

Ketones..... Diacetyl, Acetone } in the forerun
Methyl Heptenone (0.2–0.3%)
Furthermore, traces of:
A ketone or aldehyde; semicarbazone m. 229°–231° (see above)
α,β-Dihydropseudoionone

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Sesqui-terpenes...	Mostly bicyclic (ca. 1%)
Diterpenes... (2–3%)	α-Camphorene A bicyclocamphorene(?)

Recently Naves [40] reported that the oil of lemongrass distilled from *Cymbopogon flexuosus* Stapf in Guatemala contains about 0.7 per cent of a *diterpene ketone* $C_{20}H_{30}O$ which combines with the P reagent of Girard and Sandulesco under the usual conditions.

Purified by distillation the ketone exhibited these characteristics:

$b_{1.7}$.....	157°–158°	α_D.....	Inactive
d_4^{20}.....	0.9496	n_D^{20}.....	1.52060

The ketone could be oximized only with difficulty, even on boiling; it reacted very slowly with sodium bisulfite. Naves did not succeed in obtaining a semicarbazone. A dinitro-2,4-phenylhydrazone m. 128°–128.5° gave no satisfactory analysis.

As can readily be seen, the lemongrass oils are of a qualitatively complex composition. According to Naves,[41] this explains the noted differences in the olfactory value of the citrals isolated from the oils, even when purified up to a 100 per cent aldehyde content. It is somewhat difficult to free the citral entirely from other aldehydes and ketones which accompany it.

It has occasionally been asserted that the West Indian type of lemongrass oil is qualitatively inferior to the East Indian type, due to its poorer solubility. This is no criterion, however, owing to the ease with which myrcene and its condensation products may be eliminated. Lately large quantities of the West Indian type of lemongrass oil possessing a high content of citral have been produced in Central America and in some of the West Indian Islands, particularly Haiti. During World War II the American manufacturers of natural isolates and synthetics were dependent almost entirely upon the West Indian type of oil, and much progress both in the production of the oil and in the methods of isolating the citral has been made. With proper precautions in the process of extraction the citral derived from the West Indian lemongrass oil is equally as good as that from the East Indian.

Lemongrass Oil from Guatemala

Development.—The origin and development of the lemongrass oil industry in Guatemala closely parallel those of the citronella industry in the same country (cf. the monograph on "Citronella Oil from Guatemala"). Both

[40] *Perfumery Essential Oil Record* **39** (1948), 346.
[41] *Parfums France* **9** (1931), 71.

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plants were introduced in Los Cerritos by Mr. Julio Samayoa during World War I on an experimental basis. These plantations, later taken over by Mr. René Keilhauer, were subsequently greatly enlarged by his son, Mr. Minor Keilhauer. The industry reached its peak of production during World War II, when shipping difficulties prevented imports of the East Indian oil to the United States and other countries of the Western Hemisphere. At that time, America had to rely almost entirely upon the lemongrass oil produced in Guatemala (and Haiti).

The following production figures [42] will give an idea of the remarkable development of the Guatemala lemongrass industry:

	Pounds
1938	29,500
1939	63,915
1940	116,229
1941	139,751
1942	164,423
1943	190,542
1944	184,908
1945	109,265
1946	109,423
1947	129,977
1948	142,000
1949	135,000

In the course of World War II many new producers entered the field, as the then prevailing high price of oil (up to $5.00 per lb.) permitted good profits. Since then, however, prices have declined as a result of the reappearance of the East Indian product on the market. The bulk of the Guatemala oil is produced today by three large-scale operators, located in Los Cerritos, Retalhuleu, and San Felipe. A number of the smaller growers will probably lose interest in lemongrass, when prices no longer cover operating expenses.

Producing Regions.—In Guatemala, lemongrass oil is produced near the Pacific Coast, in the Departments of Retalhuleu, Suchitepequez, and Escuintla. The total acreage planted with lemongrass in Guatemala at the beginning of 1950 was 1,200 hectares.

Climate, Soil, and Altitude.—Lemongrass grows well at altitudes ranging from 400 to 2,000 ft. above sea level. The plant requires a tropical climate, abundant sunshine, and occasional heavy rains. It is not so much affected by climatic conditions as the citronella plant. Growing in much poorer soil, it can be planted in many places where citronella would not thrive. In general, lemongrass is a hardier plant than citronella and more resistant to

[42] The author is greatly indebted to Mr. Carlos Schaeuffler of Retalhuleu, one of the largest essential oil producers in Guatemala, for much of the data contained in this monograph.

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drought. In Guatemala the lemongrass fields are not irrigated, and no fertilizers are used.

Planting, Cultivating, and Harvesting.—In Guatemala the lemongrass plant (which is of the West Indian type—*Cymbopogon citratus* [D.C.] Stapf) never flowers; propagation, therefore, has to be effected by root division. The stool of a full grown plant yields ten segments. For replanting, each segment is cut to a length of about 2 ft. At the beginning of the rainy season (in May), the segments are set out 3 ft. apart, in rows also 3 ft. apart. Three to four segments are planted into each hole, which should be about 6 in. deep. Deeper planting is dangerous because the plants may develop root rot during the rainy season.

The leaves can be cut for the first time eight months after planting, and then every three months, which permits four annual harvests. Except for weeding and cleaning of the fields, not much cultivation is necessary. The plants are cut by hand with machetes—a practice involving a great deal of labor and constituting the largest item in the total cost of the oil. After four years, the productivity of a lemongrass field declines to such a point that the planting should be renewed.

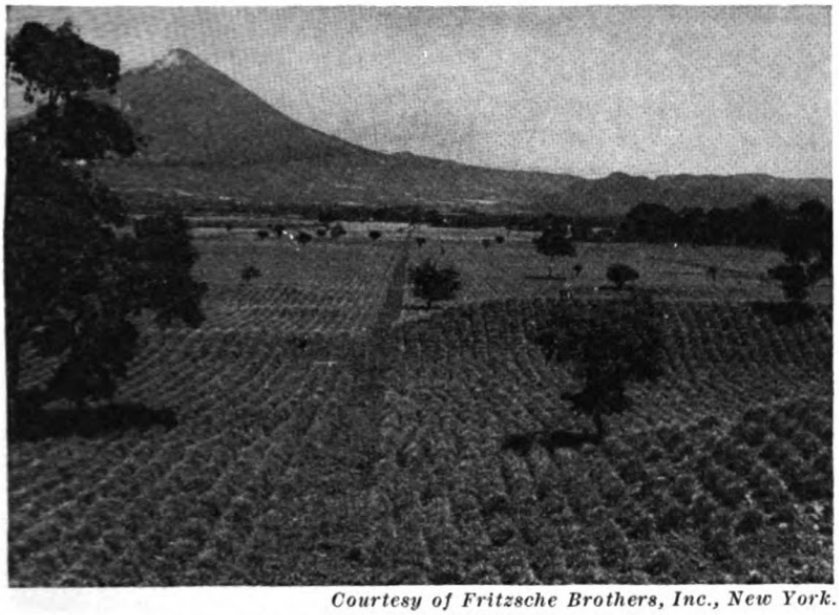
Recently, mechanized methods of farming have been introduced on certain large estates devoted to the cultivation of lemongrass and citronella. Distilling equipment also has been modernized.

Distillation and Yield of Oil.—In Guatemala, lemongrass is usually distilled while still fresh and green. The leaves are cut in the morning, then bundled and hauled to the distilleries to be distilled in the afternoon. As a result, some of the cut grass is half dried, but nevertheless still green.

Prior to distillation, the leaves should be reduced to short lengths in specially constructed, heavy-duty ensilage cutters. This facilitates charging of the stills.

The stills are of the same type as those employed for the processing of citronella (cf. the monograph on "Citronella Oil from Guatemala"). Distillation time is somewhat shorter than in the case of citronella. Prolonged distillation yields oils of low citral content; short distillation gives oils high in citral. During the rainy season the citral content of the oil is generally lower than during the dry season.

According to information given the author by the Oficina Controladora de Aceites Esenciales, Guatemala, C. A., in commercial production, the yield of oil per acre per year declines with the age of a field. Figures furnished by the Oficina in 1950 indicate that, with 3 or 4 cuttings per year, annual yield of oil per acre (in large-scale production) was 96 lb. in the first year, 74 lb. in the second, 66 lb. in the third, and 48 lb. in the fourth. Average yield of oil per acre per year was thus 71 lb.



Courtesy of Fritzsche Brothers, Inc., New York.

FIG. 6. Production of lemongrass oil in Guatemala. View of a large plantation.

Courtesy of Oficina Controladora de Aceites Esenciales, Guatemala, C. A.

FIG. 7. Production of lemongrass oil in Guatemala. View of the modern overhead irrigation system with rotating sprayers, being introduced at present on many plantations of Guatemala's Pacific Coast.

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In experiments conducted by Loustalot and Pol,[43] East Indian lemongrass outyielded the West Indian in terms of fresh grass produced, but the yield of *oil* per acre was greater in West Indian grass cut at maximum and low height because the percentage of oil was higher. East Indian grass cut at medium height yielded more oil than West Indian grass cut at the same time, because the percentage of oil was about the same and the East Indian variety yielded more grass. The average annual yield of oil per acre from plots of West Indian grass cut ten, seventeen, and nineteen times over a three year period was 131.0 lb., 132.3 lb., and 110.5 lb., respectively. The average annual yield of oil per acre from East Indian grass harvested at a height of 2.5 ft. was at least 100 lb. more than when the grass was cut at maximum or at low height. The percentage of oil was consistently higher in the West Indian grass, but there was no marked or consistent difference in citral content between the two varieties or among the three harvest treatments.

Use of Residual Leaf Material.—Recently one of the largest lemongrass oil producers in Guatemala erected a factory for the purpose of converting the spent leaf material (after distillation) into paper. Obviously, this will mean a substantial economy in the production cost of the oil and enable the producer to compete more easily with the low-priced East Indian oil.

Physicochemical Properties.—The Guatemala lemongrass oil has usually been of excellent quality, a result of the efficient method of distillation generally employed in that country. The properties fall within the limits of the West Indian type of lemongrass oil.

Numerous shipments imported and analyzed by Fritzsche Brothers, Inc., New York, had properties ranging within these limits:

Specific Gravity at 15°/15°	0.876 to 0.888
Optical Rotation	−0° 4′ to −1° 8′
Refractive Index at 20°	1.4830 to 1.4877
Aldehyde Content (Bisulfite Method)	75% to 81.5%
Solubility	Hazy to cloudy in 1 vol. and more of 80% alcohol, followed by separation of polymyrcene

Samples of oils containing less than 75 per cent of aldehydes (citral) have been examined. However, all contracts today are written on a minimum citral content (bisulfite method) of 75 per cent. This applies to the average citral content of all drums included in the shipment. A discount is allowed on any shipment with an average citral content of less than 75 per cent.

[43] *Agron. J.* **41** (1949), 375. *Chem. Abstracts* **43** (1949), 8448.

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Export.—Guatemala lemongrass oil is now exported through the Oficina Controladora de Aceites Esenciales, Guatemala City, an association founded in May 1948 and acting as consolidated shipping agency. It handles all essential oils produced in Guatemala. (For details see the monograph on "Citronella Oil from Guatemala").

Lemongrass Oil from Haiti [44]

As in most tropical countries where French is spoken, in Haiti lemongrass is commonly called "citronelle." Nothing is known about the introduction of the plant to the island. Before 1937 no plantations existed, and lemongrass was simply one of the scattered aromatic plants that thrived in the gardens of peasants and were used chiefly for the making of infusions supposed to cure tropical fevers. However, since 1937 (particularly during World War II), regular plantations of lemongrass have been started. These now cover more than 2,000 acres. The tendency is toward a further increase, despite the substantial decline in the price of the oil since the end of the war.

In Haiti oil of lemongrass, like sisal, cannot be produced by small growers and peasants. Only large-scale planting, intelligent field management, proper fertilization, and mechanized agricultural methods enable the producers to withstand heavy price fluctuations on the world market and to compete with the oil from East India. Consequently, lemongrass has never been grown outside of the plantations owned by large producers.

All existing fields are located near the distilleries. Plantations have been established and are in actual production at Ducis, Maniche, Fonds des Nègres, Hinche, and St. Michel de l'Attalaye. Experiments with plant selection, fertilization, mechanical cultivation and harvesting by means of specially constructed harvesting machines are being conducted. Although labor in Haiti is (seemingly) cheap, the cost of production is nevertheless high; and at a price of $1.00 per pound of oil, c.i.f. New York (1948), the distillers can hardly compete with the East Indian oil, unless they resort to modern field management and mechanized agriculture.

In Haiti the lemongrass plant yields a normal oil, provided it is grown in good soil, up to 1,500 ft. altitude. At high elevations (which usually receive more rainfall than the low altitudes) the factor of sunlight has to be considered, as this influences the citral content of the oil. At high altitudes the plants should be set out in rows 42 in. apart in one direction, and 36 in. apart in the other. The orientation should be east-west, to obtain a maximum of sunshine.

The grass is ready for cutting when two-thirds of the leaves have fully matured, i.e., when the tips have begun to dry out. A newly established plantation can usually be harvested for the first time eight months after planting. If the field is irrigated subsequent cuttings may take place every three or four months. In general, an irrigated plantation permits three harvests per year, whereas a nonirrigated field gives only two crops per year. The yield of grass per acre depends upon the fertility of the soil (particularly its nitrogen and potassium content), the number of weedings, the amount of rainfall, and the height to which the plants were cut during the preceding harvest. The percentage of citral in the oil is influenced by the amount of sunshine and the degree of plant maturity. Young plants or young shoots should never be distilled; they may yield more oil than older plants, but the oil will be deficient in citral.

Lemongrass represents one of the most soil-exhausting grasses known; it requires intelligent management to maintain a field in good production more than four years. Aside from nitrogen, potassium is the most important growth element.

After cutting, the grass is left in the fields for one day to reduce the moisture content. Drying for one day does not reduce the yield of oil, yet greatly facilitates loading of the trucks and packing of the grass (leaves) into the stills.

Prior to distillation the grass is chopped up in an ensilage cutter and then tramped into the retorts to prevent steam channeling. In Haiti distillation is conducted in one of the most modern tropical essential oil distilleries, with direct steam of constant pressure of 40 lb. per sq. in. (measured in the steam boiler). Water condensed within the retort is drawn off and eliminated after completion of each batch, which to distill requires about 3 hr. One metric ton of grass yields about 1 gal. of oil, a yield of about 0.35 per cent. One particular distillery equipped with 16 stills of 2.5 metric tons capacity each, and using a steam boiler of 300 h.p. capacity, can produce 5 drums of oil per 24 hr. Fifteen trucks are required to haul the cut grass from the fields and to carry the exhausted grass back to the fields for fertilizing purposes. On an average, the grass from 700 acres can be processed in a month. If not used for petitgrain, vetiver, or citronella, the distillery is then shut down until the next lemongrass crop is ready.

After completion of one charge, the exhausted grass is removed from the retort by hand, or by means of an electrically driven hoist, and dumped into trucks that haul it back to the fields or to the compost pit. When spread

months for decomposition, and the subsequent crop does not get the benefit of all the plant food that the decaying grass contains. Therefore, in the first ensuing crop, the exhausted grass acts only as a mulch, the role of which in the tropics is well known and appreciated, particularly during periods of low rainfall.

Total Production.—The following statistics give a good indication of the development of Haiti's lemongrass oil industry from 1941 to 1949 inclusive:

Fiscal Year	*Quantity of Oil in Kilograms*
1941–1942	2,093
1942–1943	6,303
1943–1944	2,968
1944–1945	4,743
1945–1946	8,071
1946–1947	23,903
1947–1948	33,422
1948–1949	24,175

Production could be increased substantially should demand and prices warrant it, and one day Haiti may easily rank among the largest lemongrass oil producing countries of the world.

Physicochemical Properties.—The Haitian lemongrass oil has properties characteristic of the West Indian type. The bulk of the oil, on analysis, exhibits a citral content higher than 76.5 per cent.

Numerous lots imported and examined by Fritzsche Brothers, Inc., New York, have had properties varying within these limits:

Specific Gravity at 15°/15°	0.883 to 0.898
Optical Rotation	−0° 8′ to −0° 27′
Refractive Index at 20°	1.4842 to 1.4888
Aldehyde Content (Bisulfite Method)	73.5 to 81.5%
Solubility	Cloudy up to 10 vol. of 80% alcohol, followed by separation of polymyrcene

Regarding the aldehyde (citral) content of commercial shipments, see also the monograph on "Lemongrass Oil from Guatemala."

Lemongrass Oil from Honduras

During World War II efforts were made to produce oil of lemongrass in the lowlands of Honduras (C. A.), near the Atlantic seashore. Almost 50,000 lb. of oil were in fact produced from 1942 to 1947. The reappearance of the East Indian oil on the North American market then caused such a sharp fall in prices that most of the lemongrass acreage in Honduras was converted to the growing of other, more profitable crops. Moreover, when five years

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old, the grass had declined to a point where harvesting became almost uneconomical. This was accompanied by a decline of the citral content of the oil from the required 75 to 70 per cent.

The whole venture in Honduras cannot be considered too successful, perhaps for the reason that the grass was planted in unsuitable soil. Throughout the years of actual production and experiments, which were carried out carefully and systematically, the yield of oil per acre was low, ranging from 12 to 72 lb. per cutting in extreme cases, and from 30 to 50 lb. in most cases. The yield of oil per acre and per year was 86 to 172 lb. with three annual cuttings, and on the average about 140 lb. The grass was cut only three times a year, because the slight increase in oil obtained by harvesting every two or three months was not sufficient to counterbalance the additional cost of harvesting.

The yield of oil per acre gradually increased as the grass became better established, but it was low whenever the grass was cut during heavy rains.

It was found that the grass should be left in the fields to dry for 3 to 5 days prior to distillation, the length of drying time depending upon weather conditions.

Before being distilled, the grass was run through twin power cutters, and mechanically conveyed into the stills. Distillation of one batch required 2 hr. and 45 min. for complete exhaustion of the grass at 5 lb. steam pressure. The cutting of the grass and the charging of the still required an additional half hour.

The yield of oil varied from 0.23 to 0.58 per cent.

The citral content of the oil ranged from 70 to 82 per cent.

Shipments of lemongrass oil from Honduras, received and examined by Fritzsche Brothers, Inc., New York, had properties varying within these limits:

Specific Gravity at 15°/15°	0.877 to 0.885
Optical Rotation	−0° 8′ to −0° 21′
Refractive Index at 20°	1.4831 to 1.4858
Aldehyde Content (Bisulfite Method)	75 to 82.5%
Solubility	Cloudy in 1 vol. and more of 80% alcohol

As regards the citral content of commercial shipments, see the monograph on "Lemongrass Oil from Guatemala—Physicochemical Properties."

Before concluding this section it should be mentioned that a sort of dermatitis appeared on the hands of laborers who handled wet lemongrass. Gloves were tried for protection, but decomposed after a few hours' use. Eventually a cold cream proved very effective in curing existing skin eruptions and in preventing further outbreaks.

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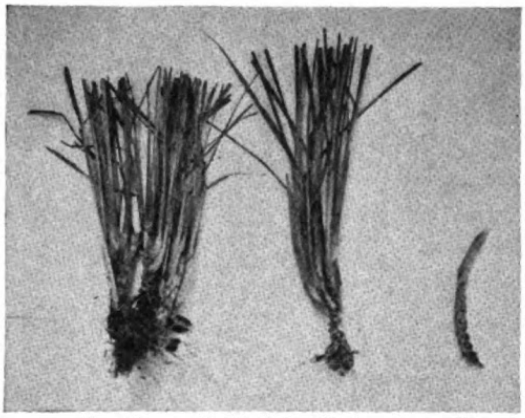
Lemongrass Oil from Puerto Rico

In 1935 the Experiment Station of the U. S. Department of Agriculture in Mayaguez, Puerto Rico, began an extensive investigation of certain aromatic plants, among them lemongrass. Many factors important in the successful production of lemongrass oil have been thoroughly studied—for example, the influence of climate, soil, light, propagation, harvesting methods, damage by insects and diseases, and methods of distillation. The results of this work have been published by Jones and Arrillaga.[45] Space permits citation of only a few points as summarized by these authors:



Courtesy of United States Department of Agriculture, Experiment Station, Mayaguez, Puerto Rico.

FIG. 8. Division of a lemongrass stool into pieces for propagation. Left, whole stool after the removal of leaves. Center, part of the stool in the process of being divided. Right, one of the many "seed pieces" obtained.

1. The grass should be propagated by division of stools and planted 4 to a hill every 2 ft., in rows 3 ft. apart. Planting is best done during the rainy season.
2. Lemongrass thrives in a well-drained soil; it can be grown on hillsides as a soil-conserving grass.
3. Lemongrass responds to fertilization with compost, manure, nitrogen, and potash.
4. The plant should not be shaded.
5. Lemongrass is best cut about four times a year at a height of 2½ to 3½ ft.
6. The grass (leaves) can be harvested at the rate of 20 to 50 tons per acre per year.
7. Prior to steam distillation the fresh leaves should be chopped.

In the author's opinion, those suggestions of Jones and Arrillaga which concern distillation with high-pressure steam, particularly, cannot be applied to industrial production without having first been tried out on a large scale. The cost of specially constructed stills, and other economic factors, will have to be considered. It should be kept in mind that the findings of Jones and Arrillaga, most interesting and detailed though they are, have

[45] "Experiments with Lemongrass in Puerto Rico," *Fed. Expt. Sta., U. S. Dept. Agr., Mayaguez, Puerto Rico* (Publication forthcoming).

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been the result of work on a small experimental scale, carried out chiefly in pilot stills.

Puerto Rico has not yet produced commercial quantities of lemongrass oil. Nevertheless, the Agricultural Experiment Station in Mayaguez has rendered a service of incalculable value to the whole essential oil industry of America, by freely distributing planting material to Central and South American countries during World War II. This enabled several countries to develop into large producers of lemongrass and citronella oil at a time when the Western Hemisphere was almost entirely cut off from its former supplies in Asia.

A small shipment of lemongrass oil produced experimentally by the Agricultural Experiment Station in Mayaguez and examined by Fritzsche Brothers, Inc., New York, had these properties:

Specific Gravity at 15°/15°	0.902
Optical Rotation	−0° 12′
Refractive Index at 20°	1.4880
Aldehyde Content (Bisulfite Method)	74.8%
Solubility	Soluble in 0.5 to 1 vol. of 80% alcohol, cloudy with more

The oil was of excellent quality; it had no odor of methyl heptenone.

Lemongrass Oil from Florida

The first experiments toward the production of lemongrass oil in the United States (in the Everglades of Florida) were made by Hood.[46] Almost twenty-five years later the United States Sugar Corporation started production on a commercial scale near Clewiston, Florida. The venture flourished for a short time because, during the early years of World War II, prices of the oil reached about $4.00 lb. Moreover, the East Indian product was not easily available, and production in Guatemala and Haiti was still limited. The American oil, therefore, found a ready market.

The lemongrass industry of Florida was based upon the principle of utilizing the spent (distilled) grass as cattle feed. The spent grass was chopped, dehydrated in an Arnold dehydrator, and then sweetened with cane molasses supplemented with a small quantity of protein concentrate, such as soybean or peanut meal. This feed permitted producers to "finish" large numbers of range steers to a high degree, without the use of any grain whatsoever. In the whole program the essential oil was, therefore, merely a by-product. To off-set the high cost of labor, Florida's lemongrass industry had to be established from the beginning on a highly mechanized basis. The

[46] *U. S. Dept. Agr., Bull.* No. 442 (1917), 1.

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reader interested in details will find valuable information in several papers by Dr. B. A. Bourne,[47] who directed the whole project. The venture was not successful, however, for the simple reason that, in the course of a few years, the cost of production more than doubled through increased costs of labor and materials. These increases were not adequately compensated for in the sales price of the oil. On the contrary, after World War II, the price of lemongrass oil declined to about $1.00 lb., and production in Florida was abandoned.

Nevertheless, it may be of interest to report the yields of oil obtained in the low organic, sandy soil on which the grass was grown in Clewiston. According to Bourne,[48] the following yields, obtained under curtailed cropping, are representative of average yields from one annual harvest, under conditions prevailing near Clewiston:

Cropping Period	*Acres Harvested*	*Average Yield per Acre*		*Pounds of Oil per Ton of Grass*
		Tons of Grass	*Pounds of Oil*	
July 1, 1942, to June 1943.....	1908.1	5.18	29.017	5.603
July 1, 1943, to June 7, 1944.....	1138.8	6.59	30.107	4.570

A sample of Florida lemongrass oil examined by Fritzsche Brothers, Inc., New York, had these properties:

Specific Gravity at 15°/15°.....	0.887
Optical Rotation.....	Inactive
Refractive Index at 20°.....	1.4871
Aldehyde Content, Calculated as Citral (Hydroxylamine Hydrochloride Method).....	72.6%
Solubility.....	Incompletely soluble in 70%, 80% and 90% alcohol

Lemongrass Oil from Brazil [49]

During World War II, Brazil became a producer of substantial quantities of lemongrass oil, West Indian type, most of which has been absorbed in Brazil itself, Argentina, and neighboring South American countries. Substantial quantities have also been exported to the United States and to Great Britain. Chief producer of the oil is the Companhia Agricola Fazendas Paulistas, a large estate located near Mattão, State of São Paulo, and cover-

[47] *Drug Cosmetic Ind.* **49,** September (1941), 262, 271. *Chemurgic Digest* **4,** No. 8 (April 1945), 148. Cf. Bourne and Nielsen, *Florida Cattleman Dairy J.* **7,** No. 12 (1943), 15.
[48] *Ibid.*
[49] The author is greatly indebted to Mr. Charles Henderson, of Cochabamba, Bolivia, for much of the information contained in this monograph.

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ing more than 130,000 acres, most of which is devoted to the growing of coffee, cotton, and to the raising of cattle. This estate (British controlled) is one of the largest single coffee-producing enterprises in the world.

Experimental work on the production of essential oils was started in 1937 and has been conducted scientifically and systematically. At the time of the author's visit to the estate, a few years ago, several hundred acres were under lemongrass cultivation. This acreage has since been increased. The future development will depend obviously upon the demand, and particularly upon the price that oil of lemongrass brings on the world market. Potential production of the oil in the State of São Paulo is large.

Most of the plantations are located within a few miles of the centrally located, high-capacity modern distillery. The pH of the soil averages about 5.5. The soil is of light sandy consistency, subject to much leaching. Provided rain falls at reasonable intervals, acidity in the soil does not seem to affect either the yield or the citral content of the oil.

New plantings are started as early in the rainy season (November to February) as possible, in order to take advantage of the cool weather. Experiments indicate that plants spaced 50 cm. apart, in rows 50 cm. apart, develop more satisfactorily than plants spaced 50 cm. x 100 cm. Planting is done by men, women, and children (who must be older than 14 years, as the law does not permit employing younger children). Clumps of planting material are hauled to the site, and the tops of the grass are cut off to within 6 in. of the root. The latter is divided, and the lower brown sheath ripped off to uncover young roots. Holes 4 to 5 in. deep are punched into the ground with a crowbar or mattock, along marked lines. Baskets of prepared roots are carried along the line, and one or two roots placed into each hole. Men then tramp each root firmly into the ground.

The young plants develop rapidly and may be cut for the first time some three months after planting. Although an early first harvest gives a very low yield of grass per acre, it seems to produce a more rapid growth as well as an increase in the number of new shoots. On older fields three harvests can be obtained per year. During the season of growth (November to April) the grass may be cut within 45 days of the previous harvest. During the dry season the grass tends to dry up; but even grass (leaves) cut in this condition appears to give a good quality of oil, with a high citral content.

The grass is generally harvested wh[redacted]

as 2 metric tons of fresh leaves in a day's work. The price contract includes loading up the carts, each of which holds about 2 metric tons of grass.

Depending upon the fertility of the soil and the fertilizer used (if any), the amount of fresh leaves per running meter in the rows varies from 1½ to 5 kg. The latter quantity, however, is obtained only after very heavy fertilization, which, in view of the relatively low price of lemongrass oil, would not appear to be profitable. It has been found that best results are obtained with dressings of cottonseed meal and potash.

On reaching the distillery, the cart or lorry is passed over a weighbridge, tared, and the total daily cut credited to the harvesters. The grass is then charged into the stills by means of pitchforks, and trampled down uniformly on wire trays that are inserted as the retort fills up. Direct steam, generated in a separate steam boiler, is gradually injected into the retort during loading, to facilitate closer packing. With the proper steam pressure the plant material is exhausted in about 1½ hr. During this period, a second still is charged so that there will be no delay other than that caused by the changing of the still head from one retort to the other. The condenser valve is then switched over, and the second still brought into action, while the first is discharged and recharged with another batch of leaf material. The use of two retorts per condenser gives, in effect, continuous distillation.

The yield of oil obtained from the first harvest of a newly established plantation may be as low as 0.1 per cent. Subsequent cuttings yield from 0.20 to 0.25 per cent of oil, the percentage being calculated upon fresh grass material. As a rule, only freshly cut grass is distilled, but it may happen that during the height of the harvest the capacity of the stills does not suffice for immediate distillation of all the cut leaf material. In such an event, some of the harvested grass is left lying in the fields (where it partly dries) until the material can be hauled to the distillery.

After separation from the oil receiver the oil is delivered by the foreman of each shift to the laboratory for filtration and drying. Samples are taken from each batch until all the grass material of one field has been distilled, when the citral content of the combined batches is assayed. Bright dry oil is stored in carboys until ready for shipment.

The citral content of the oil ranges from 75 to 77 per cent (sodium bisulfite method) in oil distilled from the first harvest of a newly established plantation; subsequent cuttings yield oils assaying from 82 to 85 per cent in freshly distilled oils. The citral content varies somewhat with the season in which the grass is cut. Contrary to the general obse

Specific Gravity at 15°	0.882
Optical Rotation	−0° 13′
Refractive Index at 20°	1.4845
Acid Number	1.2
Aldehyde Content, Calculated as Citral:	
Bisulfite Method	78.3%
Neutral Sulfite Method	74.4%
Hydroxylamine Hydrochloride Method	76.5%
Solubility	Cloudy in 1 to 10 vol. of 80% alcohol
Color	Yellow

Two shipments of Brazilian lemongrass oil received and examined by Fritzsche Brothers, Inc., New York, had the following properties:

	I	*II*
Specific Gravity at 15°/15°	0.888	0.887
Optical Rotation	−0° 10′	−0° 8′
Refractive Index at 20°	1.4863	1.4872
Aldehyde Content (Bisulfite Method)	75.5%	74.8%
Solubility in 80% Alcohol	Cloudy in 10 vol.	Cloudy in 1 vol.

In the course of the last few years the above-mentioned estate in Mattão, State of São Paulo, produced the following quantities of lemongrass oil: [50]

	Kilograms
1946	4,696
1947	3,259
1948	3,440

Lemongrass Oil from Paraguay

During the years of World War II, Paraguay started to produce limited quantities of lemongrass oil. Two samples examined by Fritzsche Brothers, Inc., New York, had these properties:

	I	*II*
Specific Gravity at 15°/15°	0.891	0.901
Optical Rotation	−0° 24′	−2° 32′
Refractive Index at 20°	1.4863	1.4857
Aldehyde Content (Bisulfite Method)	80.6%	77.0%
Solubility	Soluble in 1 vol. of 80% alcohol; opalescent in 2 and more volumes	Soluble in 0.5 vol. of 80% alcohol; very slightly opalescent in 4 and more volumes

[50] Courtesy of Mr. Dan Haggard, General Manager, Companhia Agricola Fazendas Paulistas, Fazendas do Cambuhy, Mattão.

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Lemongrass oil is now being regularly produced in Paraguay by colonies of Mennonites.

Lemongrass Oil from Various Parts of Africa

Numerous efforts have been made to produce the West Indian type of lemongrass oil (*Cymbopogon citratus* Stapf) in various parts of Africa on an experimental basis. Some of these have actually led to the production of substantial quantities of oil. Tanganyika, for example, exported 10 tons of oil in 1937. Production in a given section, however, varies from year to year, depending upon prices and general market conditions, so that no exact figures can be given.

According to Coomber and Cosgrove,[51] the citral content of lemongrass oil depends upon several factors, chiefly conditions of soil and climate, the time and number of yearly harvests, and the method of distillation. Grass cut six times a year will yield an oil with a somewhat lower citral content than oil from grass harvested only four times per year. The reason is simply that the plants probably are not fully matured when cut too frequently, and that the soil will soon be exhausted by six harvests every year. The method of distillation, too, must be rigidly controlled; higher pressures of steam (about 20 lb. per sq. in.) increase the yield of oil but decrease its citral content in comparison with distillation carried out with steam at lower pressures of about 10 lb. per sq. in. Another point is that the grass should be cut after a spell of dry weather.

Tanganyika (East Africa) for years has been producing substantial quantities of lemongrass oil. Shipments received and examined by Fritzsche Brothers, Inc., New York, had physicochemical properties varying within these limits:

Specific Gravity at 15°/15°	0.884 to 0.887
Optical Rotation	−0° 32′ to −0° 34′
Refractive Index at 20°	1.4860 to 1.4865
Aldehyde Content (Bisulfite Method)	74.0 to 78.2%
Solubility	Turbid in 10 vol. of 80% alcohol

The oils have been of very good quality.

An oil from Tanganyika investigated at the Imperial Institute in London [52] had these properties:

Specific Gravity at 15.5°/15.5°	0.8846
Optical Rotation	−0° 21′

[51] *Perfumery Essential Oil Record* **39** (1948), 351.
[52] *Bull. Imp. Inst.* **32** (1934), 216.

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Refractive Index at 20°	1.4860
Citral Content (Bisulfite Method)	81%
Solubility	Soluble in 1 vol. of 80% alcohol; turbid on further dilution

As regards solubility, the oil had the characteristics of a typical "West Indian" oil, but the content of citral approximated that of East Indian oils.

Two oils from Uganda examined in the same institute [53] had the following properties:

	I	*II*
Specific Gravity at 15.5°/15.5°	0.9178	0.8846
Optical Rotation	α_D^{20} +1° 26′	α_D^{22} −0° 13′
Refractive Index at 20°	1.4913	1.4859
Citral Content:		
Sulfite Method	72%	74.5%
Bisulfite Method		77%
Solubility in 80% Alcohol		Insoluble even in 12 vol.

The Imperial Institute [54] also analyzed an oil from the Seychelles Islands:

Specific Gravity at 15.5°/15.5°	0.9026
Optical Rotation at 19°	−0° 9′
Refractive Index at 20°	1.4884
Aldehyde Content (Bisulfite Method)	86.5%
Solubility at 15.5° in 80% Alcohol	Soluble in 0.7 vol.

Steam distilling the upper parts of lemongrass plants in Barabum, Belgian Congo, Wilbaux [55] obtained 0.507 per cent of an oil with these properties:

Specific Gravity at 15°	0.885
Refractive Index at 20°	1.4856
Citral Content (Bisulfite Method)	61%
Solubility	Soluble in 2.5 vol. of 70% alcohol

Joly [56] examined thirty-one samples of lemongrass oil distilled from plants eighteen months to two years old, grown in Upper French Ubangi, and found them to exhibit a specific gravity (d_{15}) ranging from 0.8875 to 0.8960, and a citral content ranging from 71 per cent to 75 per cent.

Five samples of oil distilled from plants which were either too young or too old had the following properties:

[53] *Ibid.*, 218.
[54] *Ibid.*, 514.
[55] *Bull. agr. Congo Belge* (1936), 295. Through *Deut. Parfümerieztg.* **23** (1937), 182.
[56] *Parfumerie moderne* **31** (1937), 27.

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Specific Gravity at 15°	0.8856 to 0.8942
Optical Rotation	−0° 10′ to +0° 5′
Refractive Index at 20°	1.4867
Aldehyde Content (Bisulfite Method)	54 to 69%
Solubility	Soluble in 0.8 vol. of 80% alcohol; soluble in 0.1 to 0.3 vol. of 95% alcohol

Two samples of oil distilled under most favorable conditions had aldehyde contents of 78 and 90 per cent.

Lemongrass Oil from the Belgian Congo

According to information furnished by various government agencies,[57] *Cymbopogon citratus* Stapf grows semiwild in all parts of the Belgian Congo. It is often erroneously called "citronella." The plant prefers a low altitude and a hot, but relatively dry, climate. Prolonged rains are harmful to lemongrass as they cause attacks by rust. The grass will grow in light soil, but commercial cultivation is profitable only in fairly fertile ground.

According to Goussens,[58] production of lemongrass oil on a commercial scale in the Belgian Congo was started in 1945 near Obokote, Ituri, 314 km. south of Stanleyville, at an altitude of about 600 m. The soil there is ancient alluvium of the lower Lualabian bed, and secondary laterite. Another area of production is located in Kama, Territory of Pangi-Maniema.

For the planting of lemongrass the ground should first be plowed 25 to 30 cm. deep. Rootstocks (which have never been segmented before) are then divided, a two-year-old stock yielding about 200 root segments. A nursery comprising 100 square meters will yield sufficient root segments to plant one hectare, calculating two root segments per planting hole. Depending upon the fertility of the soil and the manure applied, 15,000 to 20,000 segments are planted per hectare. In certain cases as many as 40,000 tufts can be planted per hectare. Planting the lemongrass between rows of coffee trees, 6 to 8 m. apart, has been tried.

The leaves (grass) are harvested with sickles. The plants can be cut for the first time six to nine months after planting, and from then on every three to four months. The number of cuttings per year, of course, depends upon the fertility of the soil and the distribution of the rainfall. In general, the grass can be harvested four times per year. A planting lasts about four

[57] Ministère des Colonies, Bruxelles. Office des Produits Agricoles de Costermansville. Institut National pour l'Etude Agronomique du Congo Belge, Yangambi. All through the courtesy of Mr. Gerard J. Danco, New York.

[58] Private communication of Mr. E. Goussens, "Plantocitra," Obokote, Belgian Congo, through the courtesy of Mr. Gerard J. Danco, New York.

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years, after which the crop has to be rotated. The following yields of green (fresh) leaf material per hectare are quoted from official bulletins:

1. With a soil of medium fertility, five harvests in three years gave 122 metric tons of plant material.
2. With a soil of low fertility, five harvests in three years gave 57 tons.
3. With a soil of medium fertility, enriched with mineral fertilizers, eight harvests in three years gave 248 tons.
4. With a soil of medium fertility, enriched with 60 tons of horse dung or 80 tons of compost, eleven harvests in three years gave 324 tons.

One experienced, large-scale producer [59] in Obokote reported an average yield of 40 metric tons of leaf material (bundled and dried in the field for two days) per hectare per year.

A medium-sized distillery comprises three stills of 2,500 liters capacity each. A still of this size can be charged with 500 kg. of whole leaves (grass), or with 750 kg. of chopped material. It is advisable to cut up the leaves prior to charging. Distillation of one batch with direct steam (3 kg. pressure per sq. cm. in the steam boiler) lasts 1 hr. The above-mentioned producer in Obokote uses stills of the tilting type (see Vol. I of this work, pp. 127 and 130) provided with water seals (hydraulic joints). Each still holds 3,900 liters. In this case distillation of one charge lasts 4 hr. During the peak of the harvest the stills are operated day and night.

The yield of oil varies greatly with the season. It is about 0.2 per cent (from chopped leaf material) in the rainy season, and 0.35 per cent in the dry season, the average yield per year being 0.2 to 0.25 per cent. Yields as high as 0.6 per cent have been reported in exceptional cases. During the rainy season the yield of oil is poor because the grass contains much moisture. Moreover, the citral content of the oil is low. On the average, one hectare yields about 40 kg. of oil per year.

In order to supply buyers abroad with lemongrass oil of high quality and of guaranteed citral content, the above-mentioned producer in Obokote submits the crude oil to rectification. A certain amount of terpenes is thus distilled off and removed. Every lot of oil is carefully analyzed, so that shipments will meet specifications of the contract. Thus, oils containing from 75 to 99.5 per cent of aldehydes (chiefly citral) are exported.

By 1949 total production of rectified lemongrass oil in Obokote amounted to about 12 metric tons per year, but an expansion to about 20 tons was planned at the time of this writing. No substantial quantities of oil were being produced elsewhere in the Belgian Congo.

[59] *Ibid.*

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In general, the lemongrass oil industry of the Belgian Congo has suffered from the scarcity of skilled labor and from the indolence of the natives. The oil is merely a by-product in the agriculture of the Congo, and only a well-managed enterprise can produce it economically. The future will depend upon the price of the oil on the world market. If demand should warrant it, the plantings could be increased largely. Moreover, the government agricultural and economic services in the Belgian Congo are anxious to open new sources of revenue, and would encourage additional plantings.[60]

The quality of the lemongrass oil produced in Obokote has been very good. Every shipment is analyzed in the laboratory of the "Office des Produits Agricoles de Costermansville."

Two typical (official) analytical bulletins quote the following figures:

Standard Quality

Optical Rotation	+0° 30′
Refractive Index at 25°	1.4857
Citral Content (Bisulfite Method)	77%

Terpeneless Oil

Optical Rotation	+0° 15′
Refractive Index at 25°	1.4860
Citral Content (Bisulfite Method)	99 to 99.5%

Shipments of lemongrass oil from the Congo, received and examined by Fritzsche Brothers, Inc., New York, had properties varying within these limits:

Specific Gravity at 15°/15°	0.880 to 0.884
Optical Rotation	−0° 13′ to −0° 17′
Refractive Index at 20°	1.4840 to 1.4850
Aldehyde Content (Bisulfite Method)	76.2 to 83.5%
Solubility	Cloudy up to 10 vol. of 80% alcohol

Lemongrass Oil from Indo-China

According to Naves and Auriol,[61] the West Indian type of lemongrass, *Cymbopogon citratus* Stapf, is cultivated also in Indo-China for the distillation of the oil, about 20 metric tons of which were produced annually before World War II. The grass is planted 50 to 60 cm. apart, in rows also 50 to 60 cm. apart, one hectare thus containing 25,000 to 30,000 plants. The planting is done by men, women, and children, the children dividing the

[60] Cf. *U. S. Dept. Commerce, Washington, D. C., Bur. Foreign Domestic Commerce,* "International Reference Service," Vol. 7, Pt. 3 (1949), No. 16—"The Belgian Congo as a Source of Crude Drugs and Essential Oils."

[61] *XVII Congrès de Chimie Industrielle, Paris* (1937).

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stools, the women preparing the segments, and the men doing the actual planting. Three or four months after planting the grass can be cut for the first time; from then on it can be harvested three or four times per year. After three or four years the planting has to be renewed.

At the Experiment Station of Ong-Yêm the following yields were obtained on steam distillation of the fresh grass: 0.60 per cent of oil from the upper parts of the grass, and 0.125 per cent from the lower parts of the grass.

The yield of oil varied with the season and the age of the plants, young grass giving the highest yield. It was lower in plants which had not been cut for a long time. One hectare of lemongrass yielded 20 to 30 metric tons of fresh grass per year. In Indo-China only fresh grass is used for distillation, field drying resulting in some loss of oil.

Distillation is carried out in stills 1.5 m. wide and 1.75 m. high, holding 3 cu. m., or 650 to 750 kg., of fresh grass. Distillation of one charge lasts about 3 hr. In a distillery located in Dôn-Thuân the average yield of oil was 0.23 to 0.35 per cent during the rainy season, and 0.45 to 0.55 per cent during the dry season. Lemongrass cultivated in Xacat on red soil yielded 0.30 to 0.35 per cent of oil, whereas grass cultivated in Ong-Yêm on gray soil gave 0.23 to 0.27 per cent of oil, corresponding to 43 to 46 kg. of oil per hectare and per year. Calculated on the basis of dry grass the oil yield was 0.8 to 0.9 per cent.

The oil derived from young plants contained less citral than that distilled from older plants.

Naves and Auriol [62] reported these properties for lemongrass oil from Indo-China:

	Oil from One-Year-Old Plants	*Oil from Older Plantings*
Specific Gravity at 15°/15°	0.878–0.894	0.882–0.902
Optical Rotation	About 0°	About 0°
Citral Content	70 to 82%	78 to 87%
Solubility of Freshly Distilled Oils	Soluble in 1 to 7 vol. of 70% alcohol	
Solubility of Oils after Storage	Soluble in 10 vol. of 95% alcohol	

Lemongrass Oil from the Caucasus

Investigating an oil of lemongrass derived from *Cymbopogon citratus* and produced in Suchum (U.S.S.R.), Rutovski, and Vinogradova [63] found that the oil contained:

Myrcene	ca. 8.5%
Decylaldehyde	ca. 2%

[62] *Ibid.*
[63] *Riechstoff Ind.* **5** (1930), 15.

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Citral *a* and *b*	ca. 55%
Geraniol	ca. 3%
Nerol	ca. 1.5%, partly as isovalerate
A Phenol and Methyl Heptyl Ketone	ca. 0.1%, with an odor of cresol ca. 0.5%, which was identified by the preparation of its semicarbazone m. 117°–118°

The same authors did not observe any methyl heptenone, limonene, or citronellal in the oil which they investigated.

According to Rutovski and Vinogradova,[64] the oils of lemongrass distilled from *Cymbopogon citratus* (yield 0.2 to 0.62 per cent) in the Caucasian Mountains generally have these properties:

Specific Gravity at 20°/20°	0.8676 to 0.9215
Optical Rotation	−2° 12′ to +12° 45′, usually inactive
Refractive Index at 20°	1.4812 to 1.4892
Acid Number	11.04 to 14.4
Ester Number	19.1 to 39.6
Ester Number after Acetylation	176.10 to 244.38
Aldehyde Content (Bisulfite Method)	51.6 to 65.0%
Solubility	Not always clearly soluble in 80% alcohol

OIL OF CITRONELLA

Essence de Citronelle *Aceite Esencial Citronela* *Citronellöl*
Oleum Citronellae

Introduction.—There are two types of citronella oil: the Ceylon type and the Java type. The former is produced almost exclusively on the island of Ceylon; the latter chiefly on the islands of Java and Formosa, lately also in Central America (Guatemala and Honduras), and on the island of Haiti.

Citronella oil came into prominence rather slowly during the second half of the last century, at which time it was produced only in Ceylon. In fact, until about 1900 the market depended almost entirely upon the Ceylon oil. Today, however, the Java type of oil is of far greater importance. Prior to World War II export figures of the Ceylon oil averaged 600 metric tons per year, while Java exported about 2,000 metric tons. World War II, the

[64] *Ibid.*

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occupation of Java by Japanese forces, and the civil war in the interior of Java after the capitulation of Japan, brought about profound changes in the production of the Javanese oil and the quantity diminished greatly. Guatemala and Honduras began to produce substantial quantities of the Java type of citronella oil, which during and after World War II found an eager market in the United States and in allied countries. At the time of this writing (1949) detailed and reliable information regarding the condition of the citronella plantations in Java is still meager. It is known, however, that in general the citronella area has been reduced to less than 8,000 hectares, because during the Japanese occupation of Java there was no great demand for the oil, and the natives suspended citronella cultivation in favor of rice, tapioca, peanuts, and other food products. Moreover, old citronella fields were neglected, some are at present partly overgrown with weeds, and the yield of oil has declined from the former 0.6–0.7 per cent to 0.4–0.6 per cent. Laborers abandoned the citronella plantings and started working in other enterprises that offered higher wages. As a result of these developments, in 1948 the production of Java citronella oil was only one-eighth of the prewar level. Nevertheless, it may be assumed with some certainty that with the arrival of more normal conditions Java will again assume a leading role in the production of citronella oil. In this respect the island is favored by several advantages: relatively low-priced native labor, a native patch-crop industry against which it is difficult to compete, the "know-how" of the native growers and distillers, and the existence of a great many distilleries, of small, medium, and large size. Ample planting material is readily available, and new fields can be in full production within one or two years, should demand for the oil and good prices encourage such expansion.

So far as the difference between the Ceylon type and the Java type of citronella oil is concerned, it should be mentioned here briefly [1] that the Java oil is much more valuable and useful, as it contains about 85 per cent of total geraniol (including at least 35 per cent of citronellal), whereas the Ceylon oil contains only 55 to 65 per cent of total geraniol (including 7 to 15 per cent of citronellal).

The Java oil serves as starting material for the extraction of several very important isolates (geraniol, citronellal) which can be converted into some of the most widely used aromatics (citronellol, hydroxycitronellal, synthetic menthol, esters of geraniol and citronellol, etc.). With an average annual

matter of fact, there is no reason why the Ceylon oil, still employed in some long established formulas, could not be replaced by the superior Java oil with its similar but stronger odor. Then, too, the Ceylon oil is often adulterated with 5 to 7 per cent of kerosene, whereas the shipments of Java oil—in normal times—are carefully checked in the analytical laboratories of the Government Station in Buitenzorg. No substandard lots are permitted to reach the overseas markets.

Botany.—The parent plant of all cultivated types of citronella is probably *Cymbopogon confertiflorus* Stapf, the so-called "mana" grass which occurs wild in Ceylon.

Citronella, a stoloniferous plant, is cultivated in at least two closely related types, which can be distinguished (a) morphologically, by the shape and length of their leaves, and (b) physiologically, by the composition of their respective essential oils:

1. *Cymbopogon nardus* Rendle, *lenabatu* (*Andropogon nardus ceylon* de Jong), the so-called "lenabatu" from which the bulk of the Ceylon oil and a very small proportion of the Java oil are derived.
2. *Cymbopogon winterianus* Jowitt, *mahapengiri* (*Andropogon nardus java* de Jong), the so-called "mahapengiri," from which the bulk of the Java oil and a very small proportion of the Ceylon oil are derived.

"Mahapengiri" can be recognized by the form of its leaves which are usually shorter and broader than those of the "lenabatu." On distillation it gives a higher yield of oil than "lenabatu"; moreover, the oil is of better quality (higher geraniol and citronellal content) than that of "lenabatu." However, "mahapengiri" requires a more fertile soil, more rainfall, and more cultural care than the hardier "lenabatu."

Oil of Citronella Ceylon

History and Development.—Perhaps the earliest note, in Europe, of Ceylon citronella oil was by Nicolaus Grimm, an army surgeon, who practiced medicine in Colombo at the end of the seventeenth century. Grimm named the grass from which the oil was derived *Arundo Indica odorata.*

The first small shipments of *"Oleum siree,"* as it was then called, arrived in Europe at the beginning of the eighteenth century. However, the oil seems to have been exported only sporadically at that time. In 1851 and 1855 samples were displayed at the World's Fairs held in London and Paris; the oil became more generally known in Europe and found increasing employment in the scenting of soaps and technical preparations. Demand for the oil grew, and large quantities were produced in Ceylon, which remained

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practically the world's sole supplier until about 1890, when Java started to produce a citronella oil of superior quality. Today the supply of the Java type of oil (produced in Java, Formosa, Guatemala and Honduras) by far exceeds that of the Ceylon type. It is only because its price is usually lower that the Ceylon oil can withstand the competition of the Java type of oil.

Botany.—The bulk of the Ceylon citronella oil is distilled from *Cymbopogon nardus* Rendle (*Andropogon nardus ceylon* de Jong), locally called "lenabatu." This plant, cultivated on a large scale in the south of Ceylon, grows to a height of about 1.5 m. Its leaves are long and slender. It is hardier, has a longer life span, flourishes in a less fertile soil, and requires less cultural care and moisture than "mahapengiri" (see above, section on "Botany," and below). However, the quality (i.e., total geraniol and citronellal content) of the oil derived from "lenabatu" is inferior to that of the oil distilled from "mahapengiri."

In addition to the "lenabatu" just discussed, Ceylon also produces a small quantity of the Java type citronella *Cymbopogon winterianus* Jowitt (*Andropogon nardus java* de Jong), locally called "mahapengiri." Commercial shipments of Ceylon citronella oil consist either of oil derived exclusively from "lenabatu," or of mixtures of "lenabatu" with "mahapengiri," in which, however, the former very largely predominates. In some localities of the citronella producing regions of Ceylon the two types of grass grow almost side by side, but the quantity of the "mahapengiri" is negligible.

It has been suggested repeatedly that in Ceylon the "lenabatu" be abandoned completely, and that the acreage be replanted with "mahapengiri," in order to improve the quality of the Ceylon citronella oil. This, however, is not possible because climatic and soil conditions are not generally favorable for the cultivation of "mahapengiri." Under the conditions prevailing in Ceylon the latter type of grass gives a very poor yield of oil.

Cymbopogon winterianus Jowitt ("mahapengiri") owes its species name to the fact that years ago a Mr. Winter, an important citronella oil distiller in Ceylon, tried to cultivate the "mahapengiri" (and for a time actually succeeded) for the purpose of distillation. Later it was replaced by the hardier "lenabatu." This development also led to the local designation "Winter's grass" or "old grass" for "mahapengiri."

"Lenabatu," also called "new grass," is, perhaps, a cross [2] between "mahapengiri"—which, according to Jowitt,[3] occurs in Ceylon in the wild as well

Producing Regions.—The citronella producing regions of Ceylon are located in the extreme south of the island. Starting near the beautiful bay of Weligama, the southern limits follow the coast of the Indian Ocean eastward toward the port of Tangalla; from the coast the section stretches north into the mountains of the Southern Province. In this area about 30,000 acres are planted with citronella. In 1946 the exact figure was 30,033 acres.[4]

Two subsections of production may be distinguished:

1. The larger and more important of these subsections stretches, in the form of a huge triangle, from Matara to Tangalla and north. Most of the grass here is grown on well-kept plantings.
2. The smaller subsection lies to the north of Weligama. The grass here grows partly semiwild on old and neglected plantings, which have not been properly weeded, with the result that more than one type of citronella grass is harvested and distilled.

The oils produced in the two subsections resemble each other closely, except in respect to specific gravity, which is occasionally lower in some batches of oil from the Weligama district.

The localities where citronella is grown are for the greater part slightly hilly and rather infertile. The annual rainfall amounts to 60–70 in., but for the larger part of the year there is very little rain.

Planting and Cultivating.—All plantings are owned by Singhalese, none being operated by European producers or exporters. The fields range from scattered small patches (about ½ acre) up to 300 and more acres on large estates, which are owned by wealthy natives. These landowners sometimes rent a part of their fields, as well as the necessary distillation posts, to small producers on a crop-sharing basis. The owner then receives one-sixth of the oil produced on the rented fields, while the sharecropper retains five-sixths; from this he must pay all expenses of cultivation, harvesting, and distilling.

Except for the small quantities of semiwild grass growing on old, neglected fields (which are not carefully weeded during years of low prices), all the grass is cultivated. Plantings are located usually on the poorer type of soil, particularly on the slopes of hills. Prior to planting, the soil is prepared by burning old grass and weeds, and by hoeing.

For planting, stools of fully grown, healthy plants are divided, and set out 1.5 ft. apart. This is done during the two rainy (monsoon) seasons, which last from the end of April to the end of June, and from middle of October to middle of December.

Depending upon the quality of the soil and the amount of rainfall, from six to eight months are required for the new plants to reach a sufficient size

[4] Agricultural census of 1946.

(3 to 5 ft. tall) to be harvested. The cutting is done with sickles, by women and girls. Three harvests a year are possible on the same field, the date of cutting depending upon the time of planting. Harvesting thus goes on throughout the year, but there are three main seasons, i.e., May to June, August to September, and December to the middle of January. The grass is never cut during rain spells, because it may ferment while lying in the fields after cutting. Moreover, the distillation of wet (or fresh) grass requires long hours and a great amount of fuel. The cut grass is therefore

Courtesy of Fritzsche Brothers, Inc., New York.

FIG. 9. Citronella oil production in Ceylon. Harvest of the grass.

allowed to dry in the fields, which requires from 1 to 3 days, depending upon sunshine and atmospheric conditions. The dried grass is then tied into bundles, stacked up in the fields for further drying, and finally hauled on bullock carts to the distilleries.

The plantings should be weeded out annually, and fertilized biannually with the ashes of citronella grass which has been distilled and burned (as fuel). Frequent fertilization encourages the growth of the plant and increases the yield of oil per acre. If well taken care of, a field of "lenabatu" may be productive for as long as twenty-five years, at the end of which time it must be replanted. In most cases, however, the yield of grass and oil diminishes substantially after the tenth year, and the field becomes unprofitable. If not properly cultivated a planting may show signs of decline after five years.

Exceptional fields are said to be more than fifteen years old. While surveying the production of citronella oil in Ceylon, the author heard of fields

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for which an age of fifty years was claimed. It seems probable that such claims represent an exaggeration.

Yield of Grass and Oil per Acre.—According to Volkart Brothers,[5] the average yearly yield of Ceylon citronella oil per acre amounts to about 50 lb. "Lenabatu" grass contains approximately 0.5 per cent of oil. The yearly yield of dried grass per acre amounts, therefore, to about 90 cwt. Other sources report yields ranging from 60 to 90 cwt. of grass per acre.

The maximum yield of a plantation is obtained in the third year, at which time it reaches 60 or 80 lb. of oil per year per acre. After the fifth year the yield diminishes. Between the fifth and the fifteenth year it amounts to only 40 lb. of oil per year per acre. After the twenty-fifth year the field is usually replanted.

The opinion has been expressed [6] that the yield, as well as the quality, of the Ceylon oil could be improved by more intensive methods of cultivation (green manuring, proper shading, etc.) and more selective methods of propagation.

Distillation.—The dried plant material is processed in numerous distillation posts distributed throughout the citronella regions of Ceylon. These distilleries, which resemble one another closely in construction, are usually located at the base of hills, where a sufficient supply of cool water is available. A typical distillation post consists of two stills connected with one and the same condenser, the latter being inserted in a rectangular water basin made of concrete and imbedded in the ground. The stills, of galvanized iron, are usually 8 ft. high and 4.5 ft. wide; a few are larger. The two stills are operated alternately—while one still is being charged or discharged, the other remains in operation. For this reason the two stills have only one helmet (with an attached gooseneck). Distillation is carried out with live steam generated in a separate and simple steam boiler, the latter partly imbedded in the ground. Warm water drawn from the condenser tank serves for feeding the steam boiler. For heating, exhausted and sun-dried plant material is used. The ashes are dumped in the fields for fertilization purposes.

The oil separator and the container holding the separated oil are housed in a locked subterranean room, accessible through an underground passage, to which only the proprietor or trusted foreman has the key.

The entire distillation post is housed within walls, and protected by a protruding roof.

Only dried plant material is used, as distillation of fresh material requires too much fuel and is said to yield an oil of less agreeable odor. During dry-

[5] Private communication of Volkart Brothers, Galle Agency, Galle, Ceylon.
[6] Cf. *Perfumery Essential Oil Record* **26** (1935), 88.

ing of the cut plants in the fields, care has to be exercised to prevent fermentation and rotting.

Distillation of one charge lasts about 6 hr. This rather protracted distillation is necessary because of the low steam pressure in the boiler, the limited capacity of the latter, and the comparatively large charge of plant material in the still.

According to the industrial census of 1946, there were in the Southern Province 321 citronella distilleries, but 64 of these distilleries were not operating in 1946.

Courtesy of Fritzsche Brothers, Inc., New York.

FIG. 10. Citronella oil production in Ceylon. A typical distillery.

Trade of Oil.—In the larger and well-managed distilleries the oil is handled carefully and stored properly. The estate owners who produce large quantities of oil may deliver their output directly to exporters, but small operators or sharecroppers usually sell their lots to field brokers. In many cases the small producers are financed by middlemen, who may make substantial profits by charging interest on the advanced funds.

The leading Ceylon exporters have agencies in Galle or Matara, purchasing copra, rubber, coir yarn, cinnamon, oil of citronella, and other products of the island from the native producers. The buying of citronella oil is by no means a simple matter, because the manager of the agency, although no chemist, is expected daily to evaluate numerous small lots of citronella oil brought in by field brokers. Unless the latter receive an immediate acceptance, they simply go next door and offer the lot to a competing purchasing agency. The analytical assays must, therefore, be simple, yet as reliable

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as possible. Determination of the specific gravity, a test for the presence (absence!) of alcohol, and a simple solubility test for the presence (absence!) of kerosene are all that can be expected of a rough and ready test for the quality of each lot. Beyond that the purchasing agent cannot possibly go.

If a lot is accepted, the field broker is paid in cash, or credited against loans advanced. If refused, he usually offers his oil to a competing exporter nearby. In the course of these transactions a good deal of oriental business craftiness is exercised: frequently intermediaries succeed in playing competing exporters one against the other, particularly if one exporter is short of oil. It is surprising how well the intermediaries are informed about the stocks, commitments, and requirements of competing exporters.

When a certain amount of oil has been purchased, the accumulated small lots are bulked in the warehouses of the exporter's branches in Matara or Galle. These large lots are then stored in tanks until the oil is sufficiently clear to permit easy filtration. Impurities settle to the bottom of the tank, and the supernatant liquid may often be so clear that filtration becomes unnecessary.

On aging, the oil undergoes certain changes: the specific gravity increases slightly, and the solubility in 80 per cent alcohol decreases. Genuine oils which contain no added kerosene (see below) retain their originally good solubility for about three months. The color also changes, particularly when the oil is exposed to air and light. The color may range from yellow to slightly green.

As a rule, the exporters standardize their bulkings, or make them up on the basis of orders from abroad. The oil is shipped in second-hand lubricating oil drums of 45 gal. capacity, and a small percentage (about 6 per cent) in galvanized iron drums. Shipping ports are Galle and Colombo, where most of the exporters have their main offices and warehouses ("Go-downs"). Some of the larger exporters operate their own very simple testing laboratories, where the outgoing shipments are submitted to a few routine tests. When there is doubt as to quality, a sample of the lot is submitted to the city analyst in Colombo. No government control is exercised over outgoing shipments of citronella oil, and purity and quality are matters of contract between exporters and foreign buyers.

Quality of Ceylon Citronella Oil.—In respect to quality, the Ceylon citronella oil industry differs considerably from that of Java, where every shipment of essential oil is submitted to a rigid analysis in the government laboratories, before an export permit is granted, any adulterated or substandard batch being rejected. To the lack of government control over exports of Ceylon oils

been adulterated with about 6 per cent of kerosene. This practice came into vogue years ago when some exporter had the "bright" idea of adding a small quantity of kerosene to the oil in order to reduce its price by a few pennies. The surprising part is that the idea took hold, with the result that the trade became used to adulterated citronella oil from Ceylon, and for many years has accepted this quality readily under the label of "ordinary" or "market" or "fair average quality" (f.a.q.). Needless to say, this practice has greatly handicapped the Ceylon oil in its competition with the Java citronella oil which, aside from its purity, offers the added advantage of a higher citronellal and total geraniol content. In the course of years, many efforts have been made, on the part of serious exporters and importers, to put a stop to this nuisance. Among other measures the establishment of government control over outgoing shipment of the Ceylon oil has been suggested, but these efforts have met with little success. Several arguments have been advanced in favor of retaining the old practice of adding kerosene to the Ceylon oil. It is asserted, for example, that the trade has become used to this type of oil and likes it because of its lower price, or that without the benefit of a lower price the Ceylon oil could not compete with the Java oil, or that government control would mean a government monopoly, etc. All of these points are obviously irrelevant: by the same logic most essential oils could or should then be adulterated, and instead of (say) 10 per cent of kerosene, any quantity could be added, to suit the purse of the buyer! It would seem more logical for the buyer to do the cutting of the oil himself, when necessary, or to use less oil in his preparation, rather than to purchase an oil cut with a neutral kerosene fraction, which adds nothing to the odor strength of the oil. Moreover, since the kerosene fractions used in Ceylon for the purpose of adulteration originate chiefly from the United States, they are shipped overseas for a long distance only to be shipped back to America or Europe. It would be more economical, under the circumstances, to save the cost of transport of the kerosene fractions and add them to the pure oil in America or Europe, if this should really be desired.

As a result of the practice of adulteration, there are now two distinct types of commercial Ceylon citronella oil:

1. The so-called "estate" oils, which are generally pure, containing no kerosene. These oils are produced [redacted]

citronella oil, but an improvement of this figure can be expected in the near future, for reasons explained below.

Serious exporters prefer to trade in estate oils, because the purchase of such pure oils in large quantities from the estates is much easier than the collection of numerous small lots (all of which have to be examined) from many intermediaries, who may not be altogether reliable. Moreover, exporters often enjoy close personal connections with the owners of large estates, and know them to be reliable suppliers of all kinds of products.

2. The so-called "ordinary," or "market," or "fair average quality" (f.a.q.) oils. They contain 5 or slightly more per cent of kerosene fractions, and are often cut down to the extreme limit permissible with the "Schimmel solubility test" (see below), upon which contracts for this type of oil are usually based. Some lots of the f.a.q. oils are produced on the larger estates, but most of the batches consist of bulkings of numerous small lots, purchased by the exporters from middlemen or field brokers. Until recently, about 70 per cent of the total annual exports of Ceylon citronella oil consisted of f.a.q. oils. It should be mentioned that the kerosene is added by the producers, but frequently, and where possible, the field brokers, middlemen, and even the exporters add some more kerosene until the limit permitted by the "Schimmel solubility test" is reached.

To put an end to the practice of adulteration of Ceylon citronella oil with kerosene, Schimmel & Co., over a period of years, introduced three simple solubility tests, which show the presence of kerosene or petroleum fractions in the oil. Details of these tests will be found below. Of the three tests, only the first, or "old Schimmel test," has been adopted by the trade, but it is powerless to detect adulteration with kerosene amounting to less than 5 per cent. Moreover, pure oils occasionally fail to pass this test. Consequently, adulterated oils continue to reach the market in large quantities, and, because of legal complications, even the U. S. Customs authorities are unable to stop the entry of such lots into the United States.

It is only recently that the Scientific Section of the Essential Oil Association of U.S.A. (E.O.A. No. 12) has established definite standards for Ceylon citronella oil, specifying that the test for the presence of mineral oil (saturated hydrocarbons) be negative. Details will be given below in the section on analysis. Although the specifications of the Essential Oil Association of U.S.A. have as yet no legal standing, they have nevertheless been of great benefit to the industry, insofar as most American essential oil houses now try to supply oils which meet these specifications. To this end,

chase chiefly estate oils. Thus, there is hope that the old practice of adding kerosene to Ceylon citronella oil will gradually be abandoned, at least as far as the imports to the United States are concerned.

Physicochemical Properties.—The odor of the Ceylon citronella oil is coarser than that of the Java oil. Its content of total geraniol (geraniol + citronellal) and citronellal is substantially lower than that of the Java oil, which fact is the principal reason for the lower quality and value of the Ceylon oil. (Cf. the monograph on "Citronella Oil Java, Physicochemical Properties.")

The color of the Ceylon oil ranges from yellow to light brown. A green color indicates the presence of copper. The copper can be removed by treatment of the oil with aqueous solutions of acids, particularly tartaric acid (cf. Vol. I of this work, p. 378).

There is no purpose in recording the limits of the physicochemical properties of Ceylon citronella oils that have been adulterated with mineral oil

	"A"	"B"	"C"	"D"	"E"	"F"
Solubility in 80% Alcohol, at 20°	Soluble in 1 vol.; opalescent in 4.5 vol. and more	Soluble in 1 vol.; opalescent in 3.5 vol. and more	Soluble in 1 vol.; opalescent in 5 vol. and more	Soluble in 1 vol.; slightly opalescent in 10 vol.	Soluble in 1 vol.; slightly opalescent with more	Soluble in 1 vol.; slightly opalescent with more

(The solubility of samples "A," "B" and "C" was determined according to the "new Schimmel test"; for samples "D," "E" and "F" the "old Schimmel test" was applied.)

Shipments of "estate oils" analyzed by Fritzsche Brothers, Inc., New York, had properties varying within the following limits:

Specific Gravity at 15°/15°	0.898 to 0.910
Optical Rotation	−9° 0′ to −18° 0′
Refractive Index at 20°	1.4790 to 1.4850
Total Geraniol Content	55 to 65%
Aldehyde Content, Calculated as Citronellal	7 to 15%
Solubility	Clearly soluble at some dilution between 1 and 2 vol. of 80% alcohol. When diluted to 10 vol., the solution should remain clear, or become not more than slightly opalescent

Analysis.—Details regarding the methods of analysis of citronella oil will be found in the monograph on "Citronella Oil Java," under the sections dealing with "Physicochemical Properties" and "Analysis." These methods apply also to the Ceylon oil. They include the so-called "oleum test" for the detection of added mineral oils or petroleum fractions, and determination of the flash point.

A few years ago the Essential Oil Association of U.S.A., Scientific Section (E.O.A. No. 12) suggested the following test for the presence of mineral oil (saturated hydrocarbons):

Place 20 cc. of fuming sulphuric acid, containing 15 per cent free SO_3 in a graduated, narrow neck Babcock bottle, 50 cc. capacity, cool in ice bath 10 min., keep bottle in ice bath and add 5 cc. of Oil Citronella Ceylon dropwise at such a rate that the bottle remains cold. Incline the bottle and rotate continuously during the addition, which should require about 5 min. When no further reaction is apparent remove from ice bath, bringing slowly to room temperature with frequent cautious shaking. Wash down neck with 3 or 4 cc. of fuming sulphuric acid.

When no further reaction is apparent on shaking, shake vigorously for 30 seconds. Place bottle in water bath and heat slowly to 60° C. with frequent agitation. (Caution is required, as escaping gas may force some solution from bottle.) Heat at 60°–65° C. for 15 min., shaking the contents carefully but vigorously 8–10 times during this period. Remove from bath and without cooling, carefully add sulphuric

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acid (specific gravity about 1.84) until the bottle is about three-fourths full. Shake well. No material should adhere to stem and sides at this point; cool to room temperature, add sulphuric acid (specific gravity about 1.84) until the level is about two-thirds up in neck of flask. Centrifuge 10 min. at 1,200 revolutions per minute or stand overnight and read, centrifuge an additional 10 min. Pure Oil Citronella Ceylon should have no readable separation.

Solubility.—These oleum tests (which, except for slight modifications, are fundamentally the same) practically supersede and eliminate the various solubility tests which were developed years ago by Schimmel & Co. for the special purpose of detecting the presence of mineral oil fractions or kerosene in Ceylon citronella oil. Details pertaining to the "old Schimmel test," the "new Schimmel test," and the "raised Schimmel test" will be found in Vol. I of this work, p. 332. As has been pointed out, the "new" and the "raised" Schimmel tests have never been adopted by the industry. The "old Schimmel test," often referred to simply as "Schimmel test," offers a rough and ready means of detecting addition of kerosene amounting to more than 5 to 7 per cent; oils containing less kerosene will usually pass this test. Many contracts for Ceylon citronella oil, particularly in Europe, are still based upon this test. It is primarily for this reason that the test is mentioned here.

A few years ago, the (British) Essential Oil Sub-Committee of the Society of Public Analysts' Analytical Methods Committee issued a report dealing with a solubility test for citronella oil.[8] The Sub-Committee found the "Schimmel test" to be unsatisfactory, because it is not sufficiently well defined to give concordant results in different hands. Hence the following test (which should be known as the "London Solubility Test") has been recommended:

One volume of citronella oil should form a clear solution at 20° C. with from one to two volumes of 80 per cent v/v alcohol, and with a total of four volumes of 80 per cent v/v alcohol; the solution should not be more than faintly opalescent and there should be no separation of oily drops on standing overnight in a stoppered cylinder at 20° C.

In practice this test is actually very similar to the "new Schimmel test" (see Vol. I of this work, p. 333) based on the description given in the Bericht Schimmel & Co. of 1923.[9] In the "new Schimmel test," 80 per cent alcohol is added drop by drop to the point of maximum cloudiness or turbidity, the mixture then being permitted to stand undisturbed to ascertain whether or not any separation of oily droplets occurs. In actual practice, the point of

is cautioned that violent shaking should be avoided to prevent the separated oil from being so finely divided that separation into droplets will not be noticed until some considerable time afterward. Such a warning might well have been included in the "London test," to hasten the routine examination of many samples in a commercial laboratory.

Total Production.—

ANNUAL EXPORTS OF CEYLON CITRONELLA OIL 1939–1948 (10 YEARS) [10]

Year	*United Kingdom*	*U.S.A.*	*India*	*S. Africa*	*Other Destinations*	*Total Tons*
1939.....	221	270	31	8	161	691
1940.....	262	138	68	4	124	596
1941.....	259	115	176	18	50	618
1942.....	275	232	157	20	112	796
1943.....	199	138	53	62	153	605
1944.....	71	321	69	44	82	587
1945.....	91	169	79	84	87	510
1946.....	412	139	58	33	128	770
1947.....	84	219	69	45	153	570
1948.....	173	134	115	51	227	700
Average 10 Years.....	205	187	87	37	128	644
Average Percentage....	31.8	29.0	13.5	5.8	19.9	100

From these figures it can be seen that during the past ten years the average export figures amounted to 644 tons per year.

The yearly production of citronella oil in Ceylon ranges normally from 650 to 700 tons. Unusual droughts may reduce this quantity occasionally by about 100 tons.

Chemical Composition.—The chemical composition of the Ceylon type of citronella oil (derived from *Cymbopogon nardus* Rendle, *lenabatu*) has not been investigated as thoroughly as that of the Java type of citronella oil (derived from *Cymbopogon winterianus* Jowitt, *mahapengiri*), large quantities of the latter being processed technically for the isolation of geraniol, citronellal and other components.

The following substances (listed approximately in order of their boiling points) have been observed in oil of citronella Ceylon:

Camphene. From the terpenes, which constitute 10 to 15 per cent of the oil, Bertram and Walbaum,[11] and Schimmel & Co.[12] isolated camphene. Conducting gaseous

[10] Courtesy Volkart Brothers, Inc., New York.
[11] *J. prakt. Chem.* [2], **49** (1894), 16.
[12] *Ber. Schimmel & Co.*, October (1899), 12; April (1912), 40.

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hydrochloric acid into an ethereal solution of the fraction b. 157°–164°, Bertram and Walbaum obtained a solid hydrochloride which, on heating with water to 100° under pressure, readily yielded camphene. Treating the same fraction with a mixture of glacial acetic acid and sulfuric acid, Schimmel & Co. prepared isoborneol m. 212°; they isolated camphene in solid form also directly by careful fractionation of the oil. According to Naves,[13] Ceylon citronella oil contains from 5 to 6 per cent of camphene (formylation by the Ljubomilov method).

Limonene. Identified by Schimmel & Co.[14] by preparation of the tetrabromide m. 104.5°, and of the nitrolbenzylamine derivative m. 92°–93°.

Dipentene. Characterized by Bertram and Walbaum,[15] and by Schimmel & Co.[16] through the tetrabromide m. 124°–125°.

Other Terpenes(?). When fractionating the oil, the last named workers [17] noted the presence also of other terpenes which had a very low specific gravity; these could not be identified (possibly myrcene was among these.—The author).

Methyl Heptenone. The forerun of the oil furthermore contains traces of methyl heptenone, which, in the course of the above cited investigations, was identified by preparation of its semicarbazone m. 134°–135°.

d-Citronellal. The aldehyde $C_{10}H_{18}O$, forming one of the main constituents of Ceylon citronella oil, has been the subject of considerable controversy (cf. the monograph on "Citronellal" in Vol. II of this work, p. 336). Dodge [18] expressed the opinion that the compound in question was an aldehyde, and suggested the empirical formula $C_{10}H_{18}O$; definite proof for its aldehydic nature was offered by Semmler.[19] Ceylon citronella oil contains 5–16 per cent of citronellal in dextrorotatory form. According to Naves,[20] the citronellal present in Ceylon citronella oil is slightly racemized: $[\alpha]_D^{20}$ +12° 6′ to +12° 36′. The statement that citronellal appears to have a very slight rotatory power, contrary to what is known of citronellal derived from Java citronella oil, is a myth (Naves.).

l-Borneol. In the citronellal fraction Schimmel & Co.[21] observed *l*-borneol, which was identified by preparation of its phenylurethane m. 138°. According to Naves,[22] the *l*-borneol contained in Ceylon citronella oil is stereochemically pure: $[\alpha]_D^{20}$ −32° 42′ (c = 20, in acetic acid); −34° 13′ (c = 20, in chloroform); −36° 43′ (c = 20, in benzene).

A Substance(?) with linaloöl odor. The same fraction was found to contain a substance with an odor reminiscent of linaloöl, which on oxidation yielded citral.

[13] *Perfumery Essential Oil Record* **39** (1948), 346.
[14] *Ber. Schimmel & Co.*, April (1912), 40. Cf. Gildemeister and Hoffmann, "Die Ätherischen Öle," 3d Ed., Vol. II, 344.
[15] *J. prakt. Chem.* [2], **49** (1894), 16.
[16] *Ber. Schimmel & Co.*, October (1899), 15.
[17] *Ibid.*, April (1912), 40.
[18] *Am. Chem. J.* **11** (1889), 456.
[19] *Ber.* **26** (1893), 2254.
[20] *Perfumery Essential Oil Record* **39** (1948), 346.
[21] [redacted]

Nevertheless, the substance in question does not seem to be linaloöl, contrary to the early opinion of Schimmel & Co.[23]

Thujyl Alcohol(?). When oxidizing this linaloöl-like substance with chromic acid, Schimmel & Co. noted, aside from citral, a compound, the odor of which recalled thujone, which gave a semicarbazone m. 179°–182°. Attempts were made, using permanganate, to oxidize the fraction b. 202°–210° (which possessed an odor of thujyl alcohol) to thujaketonic acid; these were not successful, however.

Nerol. In the fraction b. 218°–230°, after it had been freed of borneol, the same investigators [24] identified small quantities of nerol by preparation of its tetrabromide m. 116°–118°.

Geraniol. From the next fraction, which forms a large part of the oil, Schimmel & Co.[25] isolated geraniol by preparing its calcium chloride compound and regenerating the pure alcohol by decomposition of the addition product with water.

Geranyl Acetate, Citronellyl Acetate, and Citronellyl-*n*-butyrate(?). The next fraction of the oil contains geranyl acetate, accompanied by citronellyl acetate and citronellol esterified probably with *n*-butyric acid. According to Schimmel & Co.,[26] citronellol does not occur in the oil as a free alcohol, but in ester form only. Citronellol was identified by preparation of the silver salt of the acid phthalate, m. 125°–126°. According to Naves,[27] the free alcohols occurring in the oil include only small proportions of citronellol.

Methyl Eugenol. Eugenol methyl ether is present in the higher boiling fractions of Ceylon citronella oil, and was characterized by Schimmel & Co.,[28] who oxidized it with potassium permanganate to veratric acid m. 179°, and also converted it to methyl isoeugenol. The latter yielded a dibromide m. 102°.

Sesquicitronellene. Removing the methyl eugenol from the high boiling fractions by extraction with 70 per cent alcohol and redistilling the residue, Schimmel & Co.[29] obtained two fractions consisting chiefly of two dextrorotatory sesquiterpenes.

The lower boiling compound b. 270°–280° (with much decomposition), b_{15} 157°, d_{15} 0.8643, α_D +1° 28′, n_D^{15} 1.51849 which, however, still contained some oxygenated substances, apparently belongs to the aliphatic series and, according to Gildemeister and Hoffmann,[30] is most likely identical with sesquicitronellene.

This sesquiterpene was later identified by Semmler and Spornitz [31] as a natural component of Java citronella oil (cf. the monograph on "Sesquicitronellene" in Vol. II of this work, p. 83).

A Sesquiterpene(?). From the higher boiling of the two above-mentioned fractions Schimmel & Co.,[32] by careful fractionation, isolated another sesquiterpene b. 272°–275°, b_{16} 170°–172°, d_{15} 0.912, α_D^{17} +5° 50′, which, however, could not be

[23] *Ber. Schimmel & Co.*, October (1899), 17.
[24] *Ibid.*, April (1912), 40.
[25] *Ibid.*, October (1893), 12.
[26] *Ibid.*, April (1912), 40.
[27] *Perfumery Essential Oil Record* **39** (1948), 346.
[28] *Ber. Schimmel & Co.*, October (1898), 17; Oct. (1899), 18.
[29] *Ibid.*, October (1899), 19.
[30] "Die Ätherisch-

freed entirely of the lower boiling fraction (sesquicitronellene), as the difference in the boiling points was only 8°.

Farnesol. When isolating geraniol from Ceylon citronella oil, Elze [33] obtained a small quantity of a fraction b_5 150°–165°. After saponification, and on steam distillation, this fraction yielded a minute portion of an oil, which on treatment with phthalic anhydride and decomposition of the phthalate gave farnesol, b_5 145°–146°, d_{15} 0.895, α_D ±0°. According to Elze,[34] Ceylon citronella oil contains 0.2 to 0.3 per cent of farnesol, free and in ester form.

Use.—Ceylon citronella oil is used chiefly for the scenting of soaps, sprays, disinfectants, polishes, and all kinds of technical preparations where price is of prime consideration.

Suggested Additional Literature

F. Sorges, "Essence of *Cymbopogon nardus* of Sicily," *Chim. ind. agr. biol.* **16** (1940), 21. *Chem. Abstracts* **34** (1940), 3013.

Oil of Citronella Java

History and Development.—Java's citronella oil industry dates back to about 1890, when, during a hunting trip near Cheribon, A. K. J. Kaffer discovered a primitive native still in which wild growing sereh (citronella) grass was distilled. Interested in the possibilities of such an oil, Kaffer later experimented with raising the grass on his own fields. Although its origin is obscure, this grass was of such good quality that in 1890 a small planting was started in the Buitenzorg Botanical Garden. Aside from Kaffer, there were other pioneers in this field, M. Treub, and P. V. van Romburgh among them, who initiated production of the grass in western Java. A. W. K. de Jong [35] and later H. W. Hofstede [36] contributed greatly to the development of the industry by their detailed studies of Java citronella oil.

In 1899 the "mahapengiri" type of citronella, introduced from Ceylon, was first planted in Buitenzorg and then propagated widely. Kaffer extended his own plantations, which were not very far from Buitenzorg. At that time it was extremely difficult to compete on the European market with the well-established Ceylon oil. In their annual report of 1900, however, Schimmel & Co. commented very favorably on a Java oil distilled by van Romburgh from plants grown in the Botanical Garden of Buitenzorg. In 1902, there

[33] *Chem. Ztg.* **37** (1913), 1422.
[34] *Ibid.*
[35] "The Ethereal Oil Yielding Plants of the Netherlands East Indies and the Preparation of Their Oils," *Ber. van de Afd. Handelsmuseum van het Koloniaal Inst.* No. 7, 183. *Indische Mercuur* **45** (1922), 1.
[36] "Citronellaolie," *Mededeelingen van de Afdeeling Nijverheid* No. 4, Buitenzorg. Archipel Drukkerij (1928).

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were two citronella distilleries in Java, one of them, "Odorata," located in Tjitjoeroeg and owned by Kaffer, the other "Tjikantjana," in Tjandjoer. Between 1900 and 1910 the European consumers began to realize that the Java oil, being superior in quality, deserved a higher price than the Ceylon oil; also, Europe's new and rapidly growing industry of aromatic isolates and synthetics could use the Java oil to far greater advantage than the Ceylon oil. As a result, Java's citronella industry expanded steadily, and in 1919 Java for the first time surpassed Ceylon in the production of citronella oil. The banner year of 1924 saw prices higher than fl. 7 per kilogram, and from then on production increased rapidly until in 1926 Java produced twice as much oil as Ceylon. At the end of 1933, during the world-wide depression, prices fell to about fl. 1 per kilogram, but even then expansion continued until about 1,800 metric tons of oil were produced in 1934, when prices fell below fl. 0.80. This increase in production despite declining prices was a result of the even more depressed condition of all other agricultural products, which induced many small growers to turn to citronella, which was still relatively remunerative.

It might be well at this point to consider the effect which the enormously increased production had on the quality of the oil. In the early years of Java's citronella industry, all oil was distilled under peculiarly uniform conditions, dependent upon identical plant material (consisting exclusively of "mahapengiri"), identical soil, altitude, stills, and working methods. The only distilleries existing at that time were large, European-owned and managed, and processed grass grown only on their own plantations. Freshly cut grass of best quality was distilled with direct steam of relatively high pressure, and for only a short time. Hence, the oils were uniform in quality, and the physicochemical properties of Java citronella oil varied within only very narrow limits.

In the early 1900's the narrow-leafed, hardier and longer-lived "lenabatu" variety of citronella was introduced from Ceylon, probably with the idea of raising it experimentally on some of the poorer soils of Java. It was hoped that this variety, although giving a lower yield and an inferior quality of oil, might nevertheless offer a solution to the problem of opening new terrains for planting. Unfortunately, however, during the years when citronella plantings were being vastly expanded and the demand for planting material was great, the inferior "lenabatu" was propagated along with the "mahapengiri." This, of course, brought about a lowering of the quality of the Java oil which had gained such a high reputation on the world markets

1934 had still other consequences. Many Chinese, and wealthier natives, took up distillation in competition with the European producers. They had to purchase their planting material ("bibit") from existing plantations, which frequently supplied the worst quality. New plantations were started on different soils, at different altitudes, and in regions with different weather conditions. The newcomers could not afford to erect large and modern steam distilleries; they used more primitive (direct fire) stills, and often distilled leaf material purchased from native growers instead of grass grown on their own plantations. The idea was to obtain as much oil as possible from the plants without paying much attention to quality; and when prices finally fell to unprofitable levels, plantings were neglected and old fields were not renewed. It is easy to understand that buyers abroad had reason to complain about the quality of the Java citronella oil. Conditions finally became so serious that the Netherlands administration had to step in and, by enacting a number of strict control measures, save the industry which had been a source of considerable income to the colony.

Botany.—The bulk of the Java type of citronella oil is distilled from *Cymbopogon winterianus* Jowitt, *mahapengiri* (*Andropogon nardus java* de Jong), called "mahapengiri" in Java. The plants, recognizable by their broad leaves, require good soil and much care in growing and cultivating. They must be renewed after a few years, because the root stocks work themselves out of the ground. Young shoots, growing from the axillary leaves of the mother plant, develop into large clumps of grass with leaves (3 to 4 ft. high) that bend outward until their ends almost touch the ground. The plants would flower if left in the natural state. Tall plants usually contain less essential oil than normal ones.

At present, most citronella plantings in Java consist of "mahapengiri" grass, but there are still a few of "lenabatu" (which accounts for the substandard lots of Java citronella oil that occasionally reach the exporters in Java).

"Mahapengiri" is not indigenous to Java. As has been mentioned, it was imported from Ceylon in the early years of the twentieth century, when several types of citronella grass were experimented with in Buitenzorg. The wild "mana" grass, *Cymbopogon confertiflorus* Stapf, which is said to be the parent plant of all commercial (cultivated) citronella grasses, does not occur in Java.

Soil, Climate, and Altitude.—There exist in Java several varieties of wild growing sereh grass, but they are not utilized for distillation. All plant

larly its citronellal content). Uniformity cannot then be expected from oils which originate from different producing regions. For a long time citronella was considered a very sturdy plant which could get along even on poor soil. This belief caused many costly errors, because such soils, after a promising start, were quickly exhausted; poor soils do not support citronella for several consecutive years without crop rotation, rest, and ample green fertilization. It is true that even exhausted plantings yield a fair amount of oil, if other favorable factors intervene, an ideal succession of rain and sunshine, for example. As a rule, however, the yield and quality of oil from plants raised on poor soils are normal only for the first three years, and then decline rapidly.

Citronella grass grows in low as well as in high altitudes (up to 2,000 ft. and more). All kinds of soils, provided they are sufficiently fertile, suit the purpose. Rich, alluvial soil, at 600 to 700 ft. altitude, and *in a humid climate with regular rainfalls,* would probably offer most favorable conditions for good yield and quality of oil, and guarantee longevity of the plantings. At lower altitudes the plants grow very slowly during the dry season, with constant danger of dying off if cut in the beginning of, or during, that period. Heavy and loamy soils of poor texture are not suited for citronella. Neither are waterlogged soils, partly submerged, partly dry, unless they are carefully drained. According to Hischmann,[37] calcareous soils, with only a thin sand layer, yield good grass and good oils for a time, but are quickly exhausted. As soon as the roots reach the lime subsoil, the citronellal content of the oil starts declining. Hischmann believes that sandy soils, on which the grass does not grow too high, offer the natural precondition for a good oil; moreover, such soils are less afflicted with weeds. Deep sand, sufficiently rich, is often better than loamy or even fertile soil. Although the growth of the plants on such sands is meager and the quantity not large, the proportion of oil in relation to weight of leaves is more favorable. When the plants grow too quickly, the formation of oil becomes a secondary function and, after all, the leaves *per se* are only ballast. It is more advantageous to obtain a higher percentage of oil from a smaller amount of leaves because bulk increases cost of transportation, labor, and steam. Virgin soil, reclaimed from jungle, yields, like irrigated land, a very large amount of grass for the first years, to the detriment of oil formation. Such was the case in Sumatra with newly established plantings, which produced insignificant quantities of citronella oil.

Soils

Planting and Cultivating.—There are no fixed rules for laying out plantations, because of the great variations in nature of terrain, labor available, and wages.

After being cleared of trees, the land is burned over and planting holes are dug. If a planter can afford extra labor, the field, after being burned, should be gone over with a mattock. The more intense the cultivation, the more favorable will be the results. In slightly hilly ground, square fields of about 4 hectares are laid out. Wagon roads should not be forgotten. In very hilly regions, conditions are entirely different, and citronella must frequently be planted on slopes which are not suitable for rice or other crops. Sereh grass may grow quite well there, provided the soil is not too poor.

It is possible to raise citronella successfully in young rubber plantations four or five years old, with the rows of high pruned trees more than 24 ft. apart. Citronella grows in the sun and in the shade, but in the latter case few new shoots appear; the growth is retarded and, consequently, there will not be more than one yearly harvest. The blades become hard and woody, so that cutting yields too much stalk material in proportion to leaves. Shade is also detrimental to the yield of oil and its total geraniol content.

Since the grass requires a fairly large amount of moisture, planting must take place only during the rainy periods. Thus, new fields are usually laid out in December-February, i.e., during the first part of the rainy season. No grower would dare to start planting in April-October because of the risk of losing his investment during the dry season. New plants which have not developed sufficiently during the latter part of the rainy season, that is up to May, usually die early in June-September. Those that survive are subnormal.

Planting material ("bibit") for sereh grass is obtained by dividing old clumps. The best type originates from the Experiment Station in Buitenzorg. Although sereh grass does flower, it is difficult to grow it from seed. When shipping "bibit," the leaf must be torn off beforehand, and the segments dried a little in the shade. Wet planting material, that has been rained upon, spoils easily.

About ten sturdy divisions ("seed pieces") can be obtained from each clump. Calculating two divisions to each planting hole, one acre of old plants would yield material for five acres of a new planting. On good soil, the plants should be set out 3 by 3 ft. apart; the interval may be less in poorer soil. If a planting is to last only a short time, 2 by 2 ft. will be sufficient. The period between harvests should also be considered, because a longer growing time requires wider planting.

and weeding are advisable to improve the yield, and maintain the plantation. In actual practice, however, little care is exercised, and it even seems doubtful whether the expense is worth-while during periods of low oil prices.

With regular cutting at intervals of about three months, starting from the beginning of the second year after planting, a plantation should be discontinued after the fourth year, because citronella is rather soil-exhausting, and any prolonged growth would be detrimental to the soil, the plants, the yield, and quality of the oil. After the fourth year, the yield of grass and oil diminishes considerably, and from the fourth to the eighth year only 40 per cent of the initial quantity of leaves, at most, can be expected. Fields older than five years are rarely profitable. Nevertheless, one frequently encounters plantations which are renewed only when they no longer give a sufficient yield; there even exist fields which are harvested up to the twelfth year. This, of course, is quite uneconomical in most cases. The natives, who plant small patches (from one-quarter hectare to two hectares) and on cheaper ground, often discontinue sereh grass after the third year, and rotate the crop by raising peanuts or tapioca. It is a fact, however, that only poor yields are obtained when citronella is regrown later on these same fields, because this type of rotation does not rebuild the soil. For this reason native plantings seldom last longer than three years, whereas large estate plantings last from four to five years, and sometimes longer, because their soil is usually more fertile and greater care is taken in laying out the sereh fields. It is difficult to make definite statements regarding the life span of a sereh plantation. In 1939 there existed a few twenty-year-old plantings which were still good, but, in general, one-quarter to one-third of the European owned plantations consisted of fields four to eight years old.

In the years prior to World War II, the very low prices of the oil made it unprofitable to plow under old citronella fields and lay out new plantings. Changing over to the cultivation of other crops was even more costly. The financial difficulties caused by the world depression had gradually forced the large estates to turn over the growing of sereh to the natives and to rely upon the purchase of native-grown grass. As a result, the large distilleries on the old European owned estates had to process ever greater amounts of leaf material grown by the natives from seedlings which had been supplied by the estates. Once the natives had their own plants it was easy for them to continue propagating, with the result that the native plantings in the vicinity of the old estates inc

material harvested. According to Hischmann's [38] analysis of government statistics from 1932 to 1936, European owned estates then comprised far more older plantings than in former years.

When a planting of sereh grass is to be renewed, the soil must first be regenerated. The fertilizers which would seem most appropriate cannot be used in Java for several reasons. The distilled and exhausted grass serves as fuel for distillation, coal being almost unknown in Java, and wood being too expensive in many sections, or having little fuel value. Chemical fertilizers, such as are used on sugar plantations, are costly, and stable dung is not available in sufficient quantity. At one time the estates used imported fertilizers when the fields were replanted after the first four years. The result was that, while the yield of grass increased, the yield of oil remained subnormal, as compared with plantings on fresh soil.

For green fertilizers Hischmann [39] suggested using *Tephrosia candida, Tephrosia vogelii, Crotalaria,* and various mimosa varieties, because they throw off a very large amount of leaves. After these bushes have grown on a field for two years, the ground, when plowed, becomes loose and almost springy, like mossy forest soil, and recuperates beautifully. The estates tried to leave these bushes on the plantings for at least one year, but the small native growers, often on the verge of starvation, are not able to employ this method at all. They simply cannot afford to leave their ground unproductive for one year, or rather for two years actually, because a new sereh planting yields nothing for the first eight or nine months.

During the war years the government of Java could do nothing to improve the quality of the grass by selective breeding. In fact, the grass has suffered by the continued growing for many years and seems to be less hardy and less resistant to diseases than formerly. When plantings are expanded during periods of high oil prices, owners of old and poor fields offer their "bibit" to other growers for new plantings, and, since it is cheaper, inexperienced prospective planters sometimes buy such inferior planting material. Obviously, this does not help to retain the original good qualities of the plant, its resistance to disease, and the yield and quality of the oil.

Harvest.—Citronella planted in December or January can be harvested for the first time in September or October, and today the native growers generally follow this procedure. At one time, the estates waited a full year until the plant had developed its full root system, which is important for a

Courtesy of Fritzsche Brothers, Inc., New York.

FIG. 11. Production of citronella oil in Java. Harvesting of the plants on a small field.

separately, using knives for the purpose. Proper timing for the cutting is advisable, in order to prevent the grass from flowering.

It is very important to choose the most opportune time of harvesting in order to increase the yield of the planting. A too short growing period decreases the productivity, and seems to affect the plants to such an extent that a field can perish within a short time. If, on the other hand, the grass is left standing for more than three months, the lower leaves wither and considerable oil will be lost; moreover, the content of geraniol and citronellal decreases. Thus, an average between the two extremes should be chosen. However, a definite cutting period cannot always be indicated, because the climate of Java, and the rainy and dry seasons are quite irregular. The best time for cutting seems to be the moment when the stems bear six adult leaves with the seventh leaf in rolled up position. The grass should be harvested three times or, under very favorable conditions, four times a year, but since the small native growers have learned that it is quite easy to transform their grass into ready money, they make four and even more cuttings without waiting for the proper harvesting time. (If a native grower needs cash, he simply cuts from his fields as much leaf material as he requires for distillation, without giving consideration to the proper maturity of the fields.)

Western Java has its rainy season from October to February, and its dry season from May to September of which, however, only July, August, and September are actually dry. The citronella harvest is larger during the rainy season than during the dry season. If old fields are harvested at the beginning of the dry season, and on days without previous early morning dew, the plants suffer much, and the fields are soon in such poor condition that it is often necessary to plow them under in order to treat the soil for the next crop.

The practice of cutting the plants too short, especially of cutting below the knot where the leaves are implanted in the stalk, is very harmful, and involves the risk, particularly during dry weather, of the blade stalks dying off. Low cutting, practiced frequently by the native growers, has another disadvantage: many nearly oil-free parts of the blades are collected which, when distilled, diminish the yield of oil. After the grass is cut, the harvesters remove the old, dried out leaves and tie the fresh leaves in bundles so that they can be carried or hauled to the distillery.

Yield of Oil.—It is difficult to express the yield of

picul = 136 lb.) of leaves per bouw (1 bouw = 1.7537 acres) or about 35 metric tons per hectare which, taking 0.7 per cent as average yield of oil, amounts to 247 kg. of oil per hectare, or almost 220 lb. of oil per acre. (One hectare = 2.471 acres.) This figure appears very high, when compared with the estate yields of 1932 to 1936, and is probably due to the fact that in those early days the very high price of the oil permitted extensive care of the citronella fields on the estates. Hischmann,[40] in 1939, confirmed such high yields on new plantations in the second year, but only in rare cases. As a rule, the yield of oil from leaves growing profusely in rich soil is substantially below 0.7 per cent. Fields which give an exceptionally high yield of oil usually consist of sandy soils, where citronella grows not as tall and more slowly. Sereh fields four to seven years old contribute only very little to the total production of an estate, and during periods of low oil prices such old fields are not economical at all. As long as the oil prices were high, an estate could carry on with some old fields, but even then an estate consisting chiefly of old fields could not be considered a paying proposition.

The natives, on the other hand, raise their grass mostly on poor soil, which in some cases has been under cultivation with various crops for generations and without any fertilization. Their leaf production during the second and third years is, on the average, 12 metric tons per hectare and per year. Based on a yield of 0.7 per cent of oil, this amount of grass represents 84 kg. of oil per hectare, or about 75 lb. per acre. Some native growers uproot their citronella fields at the end of the third year because in the fourth year the yield is too low. Thus the planting will be productive for only slightly more than two years. Most natives, however, try to keep their fields in citronella somewhat longer, which delay results in even lower average yields.

Citronella grass grown on estates is of about the same quality as that grown by natives and contains approximately the same percentage of oil. It has been claimed that the average yield of oil per acre from estate grass is higher, but this seems quite exceptional. It is difficult to arrive at comparative figures (ratio) between the quantity of leaf material produced per acre on the estates and that grown per acre on native-owned lots. Prior to World War II the total acreage of the estate plantings was known, but even then figures pertaining to the numerous small citronella plots owned by native growers were not exact. Today the picture is even more confused. According to Hischmann's analysis of the Java Government statistics,[41] only

[40] *Ibid.* (1939), 17.

about 20 per cent of the sereh grass in 1936 was produced on large plantations, the rest being grown by small-scale, native farmers, some of whom did not distill their crops but sold them to the numerous distilleries. The average yield of oil was then 0.6 per cent, which means that, based on the 1935 statistics, 276,000,000 kg. of leaves were required to distill the oil produced in that year.

When purchasing citronella grass, the distillers base their estimates on a yield of 1 lb. of oil per 150 lb. of dried leaves. Wet material is not accepted as it requires a great amount of steam for distillation. It can be assumed with certainty that all the oil not declared by the estates as own distillation is produced from leaves grown by natives, because there exists no third producing party. Thus Hischmann [42] concluded from the 1938 statistics that 7,578 hectares of citronella grown on the estates at that time yielded only 343 metric tons of oil per year, or 45 kg. per hectare, whereas 14,608 hectares of native grown citronella yielded 1,175 metric tons of oil, or 80 kg. per hectare. In view of these figures the yield (in 1938) of 45 kg. per year and per hectare on the estates was much lower than the yield obtained in the early years of Java's citronella industry when the plantations on the estates were young, vigorous, and well kept. The statistics show clearly how the low prices brought ruin to the operations of the estates during 1932 to 1936. Compared with estate yields in former years, 80 kg. of oil per hectare of native-grown grass is not unusually high. The data published about citronella production on the estates prove that nearly one-half of their citronella acreage in 1938 consisted of fields at least four year old. Half of the citronella acreage on the estates had passed the peak of normal grass production.

There exist no data on the age of the native plantings, but it is generally known that the natives plow under a sereh field as soon as the yield of grass declines considerably, whereas the estates usually started their plantings carefully in order to secure a longer life span. For this reason the peasants generally have younger fields and obtain a better average yield. In years preceding World War II, the estates could not renew their sereh fields to the same extent as formerly, and could not spend much for the upkeep of their existing fields, suffering a good deal from the low price of the oil. This lack of care was reflected in the yield. Young fields, even though not so well kept as old ones, have a much larger grass production. This very fact partially explains the discrepancy in the production figures, the other factor being that the published areas for peasant grass have alw

fields whenever it seems advisable and much more quickly than estates would or could, in order to change to other more remunerative crops.

In 1937 almost 24,000 hectares of citronella grass were under cultivation, chiefly in western Java, especially Priangan. The estates distilled the grass in their own distilleries, and there were almost 100 such distilleries, about 12 of them buying the grass from native growers. Furthermore, there existed in that year almost 80 native distilleries, quite small, and nearly all located in Priangan. In addition, there is a native home industry, in which small and rather primitive stills are used.

The figures on yield of grass and oil given above all refer to the years preceding World War II. Since the war, the Japanese occupation and the civil strife after the war, further changes have taken place in Java's citronella oil industry. At the time of this writing (1949) comprehensive and exact data are not yet obtainable, but from general information reaching Batavia it appears that the yield of oil has declined from the former 0.6–0.7 per cent to 0.4–0.6 per cent; moreover, the total citronella acreage in western Java has been reduced to less than 8,000 hectares. According to latest reports,[43] there are now in western Java altogether 7,573 hectares of citronella plantings. Of this total acreage 4,025 hectares are old patch plantings that have not yet been restored to production; 2,963 hectares are in actual production, and 585 hectares have been replanted but are not yet in production. Of this total of 7,573 hectares 6.2 per cent is of an inferior grass variety, the rest is high-grade grass. Because of the fact that most of the fields need replanting the average production of the plantings is low. Java's total production of citronella oil in 1948 amounted to about 300 metric tons only. Based on the present total acreage of about 7,500 hectares in western Java, this would mean approximately 40 kg. of oil per hectare and year, or about 35 lb. of oil per acre and year. However, when calculating on the basis of the 2,963 hectares that are actually in production (see above), the yield of oil per hectare and year would be almost 100 kg., or almost 90 lb. per acre. All of the citronella fields are at present (temporarily ?) in the hands of the natives, the estates having been divided up. Only 25 per cent of the distillation capacity in western Java is at present utilized; the rest is idle, because of several circumstances. Main centers of production now are Tasikmalaja, Tjiamis, Tjitjalengka, Bandoeng in the Priangan, and Bantam.

Harvest and Distillation.—The leaves are usually harvested in the early morning and then dried on the ground outside of the distilleries. Drying

Courtesy of Fritzsche Brothers, Inc., New York.

FIG. 13. Production of citronella oil in Java. A native-owned distillery.

Courtesy of Fritzsche Brothers, Inc., New York.

FIG. 14. Production of citronella oil in Java. A distillery on one of the large estates.

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the quality of the oil, whereas long drying, especially in the rain, is harmful. It is claimed that the leaves should not be exposed for any length of time to sun or rain, and that the material should be distilled the same day that it is harvested. If this cannot be done, it should be dried inside the factory or under sheds or on platforms near the boiler.

The distillers prefer to distill dried leaves, because they can charge more into a still and also because the distillation of dry leaves requires less steam and fuel. During periods of continued rain the crop would have to be distilled in a wet condition and, for this reason, most distillers today stop operating in rainy weather. It would be advisable to eliminate weeds from the sereh grass during the drying process, but when oil prices are low few distillers can afford this precaution. Many distillers do not bother to cut the leaves into short pieces prior to distillation. They seem to forget that whole leaves occupy more space in the retort than chopped material, and that, because of uneven distribution in the still, steam channeling and a lower yield of oil will result. Chopping of the leaves increases the economy of a still.

The faster the steam passes through the charge, the lower is the oil percentage per kilogram of condensed steam. If, on the other hand, the steam passes too slowly through the charge, the time of distillation will be longer and the hourly yield of oil per kettle lower. As was pointed out above, most distillers today use distilled and exhausted leaves as fuel, low priced wood being available only in a few localities.

Distillation starts rapidly and slows up gradually after about two-thirds of the oil has been distilled. The average yield of oil is about 0.7 per cent; [44] it fluctuates from 0.5 per cent in the rainy season to 1.2 per cent in the dry season. A still charged with 1,500 kg. of dried, chopped leaves of good quality yields about 12.5 kg. of oil.

If an oil with a high content of geraniol and citronellal cannot be obtained in a certain locality, with plants raised from good stock, other factors such as soil, climate, season, and age of the plants must be blamed.

From the above statements it would appear that the native distillers, who raise their own sereh grass, should be able to produce a better grade of oil than those large-scale distillers who have to buy their leaf material from

tillers often dry the leaves in the sun too long prior to distillation, and extend the time of distillation until the charge is completely exhausted, which, of course, gives an inferior quality of oil with a low citronellal content.

Distillation Apparatus.—Three types of stills are employed in Java—water *and* steam stills, steam stills, and the so-called "godokkans," a sort of intermediate between the first two types.

A. Water and Steam Stills.—One of the advantages which water *and* steam distillation possesses over direct steam distillation is that the danger of steam channeling in the retort is diminished. The steam develops along the entire water surface, and consequently penetrates the charge of grass quite evenly (for details see Vol. I of this work, pp. 120, 147).

The process is now used widely by the native producers. During the early years of citronella oil production in Java, distillation was carried out exclusively with direct steam, but after 1910, when natives and Chinese entered the industry, the process of water *and* steam distillation was introduced, because stills of this type are lower priced and the process itself simple. Operation of these stills is economical; moreover, the stills are not subject to the rules of the Java government applying to steam boilers.

The water *and* steam stills are imbedded into stone hearths and are provided with a grid, which supports the grass. The water beneath the grid is heated with direct fire. One manhole on top and one on the side permit charging and discharging of the plant material. The leaves are tramped into the still quite firmly, to prevent formation of steam channels; the volume of the charge diminishes greatly during distillation. Between the still walls and the plant charge—especially in stills not properly insulated—cooling and some internal condensation may take place and the condensate, which contains dark, nonvolatile extraction matter, collects beneath the grid, at the bottom of the still, and accumulates after a number of operations. It is advisable, therefore, to draw off the water in the still (beneath the grid) after each charge has been distilled, and to replace it with fresh water for the distillation of the next batch.

The ratio of the diameter of a retort to its height should be at least 1 to 1.5 or 2.

tillation (with direct fire) can thus be recommended only if small quantities of grass are processed daily, and if the price of fuel is low, as in the case of exhausted sereh grass. Operating a retort holding 8 piculs of grass, by water *and* steam distillation, requires 3 to 4 hr., which is twice as long as would be required with direct steam distillation. Distillation of citronella with water *and* steam in very large retorts, where the steam must go a long way to penetrate the entire grass mass, gives no better a yield than distillation with direct steam.

B. Distillation in Godokkans.—Distillation in the so-called godokkans represents a modified version of water *and* steam distillation. This type of installation is usually owned by wealthier natives or Chinese. The fire hearth and the water boiler are separated from the retort. The capacity of the godokkans varies from 400 to 4,000 kg. of grass. Retorts holding more than 1,500 kg. of leaves are less economical than those holding 1,000 to 1,200 kg. Retorts of small capacity require a shorter distillation time. It has been the practice to distill a charge of 1,000 to 1,200 kg. for 5 to 6 hr., although 3 to 4 hr. would be more satisfactory. After the rapid separation of a few rather unimportant volatile aldehydes, citronellal and geraniol distill over. The yield of total geraniol, particularly citronellal, diminishes after a few hours, and from then on only the less desired compounds, such as sesquiterpenes and sesquiterpene alcohols, can be collected. Under normal conditions, the composition of the oil, after 4½ hr. of distillation, is just slightly above the limit of Contract "A," which specifies a minimum of 85 per cent of total geraniol and 35 per cent of citronellal. Evidently, then, a distillation of 5 to 6 hr. would result in an oil which does not meet these requirements.

C. Distilling with Direct Steam.—In their papers on the production of

"Citronella Oil from Honduras" and "Citronella Oil from Guatemala—Distillation.") De Jong, for instance, recommended a pressure of 3 to 4 atmospheres (measured in the steam boiler), but later the producers came to realize that such high pressure, while giving a greater yield, affected the quality of the oil adversely. Confirming this view, Hofstede actually proved by experiments that a higher yield was accompanied by a lower quality of the oil. His tests showed clearly that a high steam pressure increased the yield of the oil, but lowered the percentage of geraniol and citronellal in the oil. Instead of 3 to 4 atmospheres steam pressure, 1 to 2 atmospheres were then recommended; and after Hofstede, the distillers went even further and advocated pressures of from only ½ to 1 atmosphere (above atmospheric pressure). The loss in yield of oil obtained by the use of low pressure steam is more than compensated by a high total geraniol content of the oil. Nevertheless a few producers still prefer a larger yield to a higher quality of the oil and hence apply high steam pressure, or extend the period of distillation.

If distillation is continued sufficiently long, all the volatile oil present in the plant will distill over; such oil, however, will contain also the less desirable constituents. E[illegible]

uniform temperature and steam pressure, which should not drop during the process.

The expenses of operating a steam still are usually higher than those connected with water *and* steam distillation. Competition on part of the natives is keen, and some of the estates were forced to follow their example and install water *and* steam stills, in order to produce oils of normal quality as economically as possible. Native oil originating in some of the Priangan districts has been, and still is, of good quality. Provided the natives raise high-grade sereh grass, their oils need not be inferior to the best estate oils; in fact a few of the estate producers made the mistake of emphasizing yield at the expense of quality. Moreover, oil obtained by direct steam distillation is occasionally cloudy and must be stored, prior to shipment, in a tank containing taps at various heights, through which the clarified oil can be drawn off into drums or tins.

D. Tilting Stills.—Tilting steam stills, mounted on trunnions (see Vol. I of this work, p. 130), are not indispensable for citronella distillation, but they can be discharged (emptied) very quickly and offer much saving in labor. However, their installation is considerably more costly than that of stationary stills, and the large citronella distilleries of Java, because of sharp competition with the small operators, can hardly afford them.

Citronellal Content of the Oil.—The chief merit of the Java ty[redacted]

of this work, p. 274). Not only citronellal and geraniol, but other compounds such as citronellol, citronellol esters, geraniol esters, borneol, and sesquiterpene alcohols fall into this group. This accounts for the fact that the sum of the analytical values for total geraniol, citronellal, and nonacetylizable constituents exceeds 100 per cent. The sesquiterpene alcohols in Java citronella oil amount to about 5 per cent.

The process of distilling citronella oil from the leaves resembles that of fractionating the complete oil *in vacuo*. The principal constituents distill over approximately in the following sequence:

1. Low-boiling terpenes.
2. Citronellal.
3. A mixture of citronellal, geraniol, and various alcohols and esters.
4. Geraniol.
5. Sesquiterpenes and sesquiterpene alcohols.

From what we have said under "Cultivation," it is evident that not all citronella grasses lend themselves to fractional steam distillation for the purpose of obtaining fractions of very high citronellal content. Poor leaf material simply does not contain sufficient citronellal. According to Hischmann,[47] the factors which make for a normal citronellal content may be summarized as follows:

1. Good leaf [redacted]

content of the oil declines considerably in old fields, or on exhausted soils, although the total geraniol content may remain normal for quite some time.

8. The harvested leaves should be distilled as soon as possible, and distillation should not be pushed too far. It should be carried out under carefully controlled conditions.

At present, the state of Java's citronella oil industry does not permit adherence to all these points and, therefore, it is not possible to find throughout the producing regions the same uniform quality of oils as existed during the years prior to the industry's great expansion about twenty years ago.

We have seen that a high citronellal content is usually obtained by fractionation during the distillation of the leaves. While in Java, the author collected an oil assaying about 63 per cent of citronellal and 83 per cent of total geraniol which was said to have been obtained by distillation for only 1½ hr., without any fractionation. It was produced from leaves cut only 45 days after the previous harvest, instead of the normal 65- to 80-day

the normal oils. It was said that the removal of part of the citronellal throws an oil off its natural balance.

What the outcome of the controversy might have been is of little importance, because the occupation of Java by Japanese forces completely stopped the exports of citronella oil to Europe and America for several years.

Summary.—Let us now briefly review the fundamental changes which Java's citronella oil industry has undergone since its beginning at the turn of the century.

In the early days, almost all of the oil was produced by a few large estates which worked under similar conditions—soil, altitude, climate, planting material, apparatus, and methods of distillation. The abnormally low prices prevailing during the depression of 1931 did not even permit the large estates to cover their expenses in producing the oil. On the other hand, small

been revised several times since, and adapted to changed conditions, the most important revision being that of April 1, 1938. Highly efficient administrators as the Dutch officials are, they not only set out most energetically to remedy existing abuses in the citronella oil trade, but went much further and by practical supervision, advice, and financial help to the native growers and distillers, combined with a strict supervision of production, trained them to produce oils of normal quality.

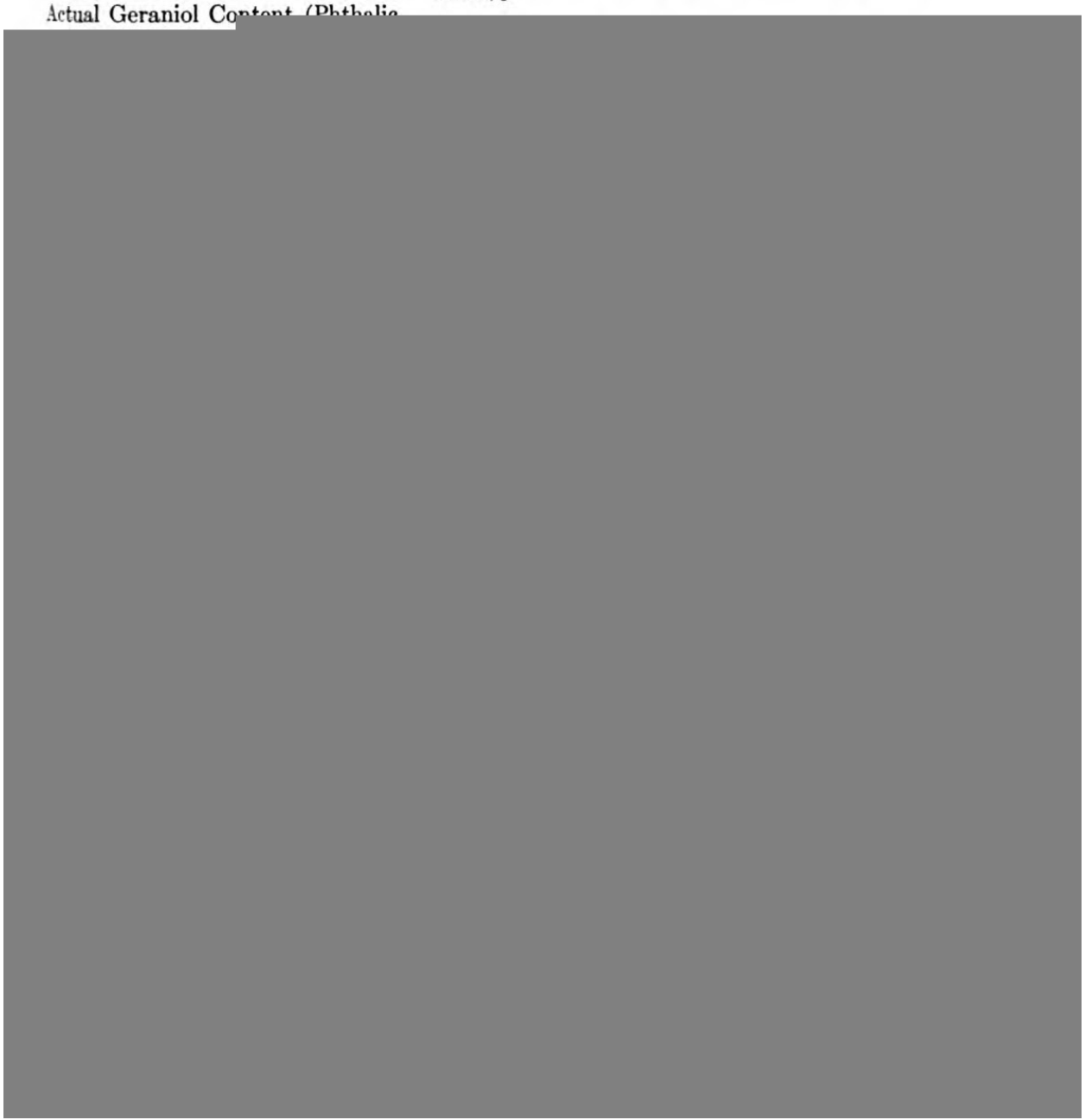
To assure the bulk of native-produced oil meeting the requirements of Contract "A" (85 per cent of total geraniol and 35 per cent of citronellal), it became necessary to raise the native sereh cultivation to a higher level by advancing credit, giving advice about planting, and supplying selected plant material for propagation. In 1935 only about 20 per cent of the exported

During the Japanese occupation, the functions of laboratories in Buitenzorg were temporarily suspended. Considerable damage was done to the Station in the course of the fighting attendant upon the liberation by Allied forces. After that, the Station for a time was occupied by adherents to the newly established revolutionary government. In these turbulent after-war years many of the shipments of oil which reached the market abroad were substandard. During the Japanese occupation metal drums were not readily available in Java, and the limited quantities of oil produced had to be stored under most unfavorable conditions and for several years. After

chemical properties described in earlier literature must therefore be accepted only with reservations. Moreover, up until a few years ago, analytical methods were imperfect and had not been standardized (as they are today).

In 1929 Gildemeister and Hoffmann [48] reported these properties:

Specific Gravity at 15°	0.885 to 0.901; oils with a high citronellal content occasionally exhibit a specific gravity as low as 0.881
Optical Rotation	Usually laevorotatory up to −4° 0′; slightly dextrorotatory oils (+1° 47′) have been observed in very rare cases
Refractive Index at 20°	1.463 to 1.475
Total Geraniol Content	Not below 85% in good oils; occasionally as high as 96%
Actual Geraniol Content (Phthalic	



Total Geraniol Content	82.3 to 89.4%, very seldom below 85%
Total Aldehyde Content, Calculated as Citronellal	28.8 to 43.9%, very seldom below 35%
Solubility at 20°	Clearly soluble at all dilutions between 1 and 2 vol. of 80% alcohol; occasionally slightly opalescent on further dilution up to 10 vol.

On the basis of analysis of a great many lots, the Government Control Laboratories in Buitenzorg (Java) established the following limits for normal Java citronella oil:

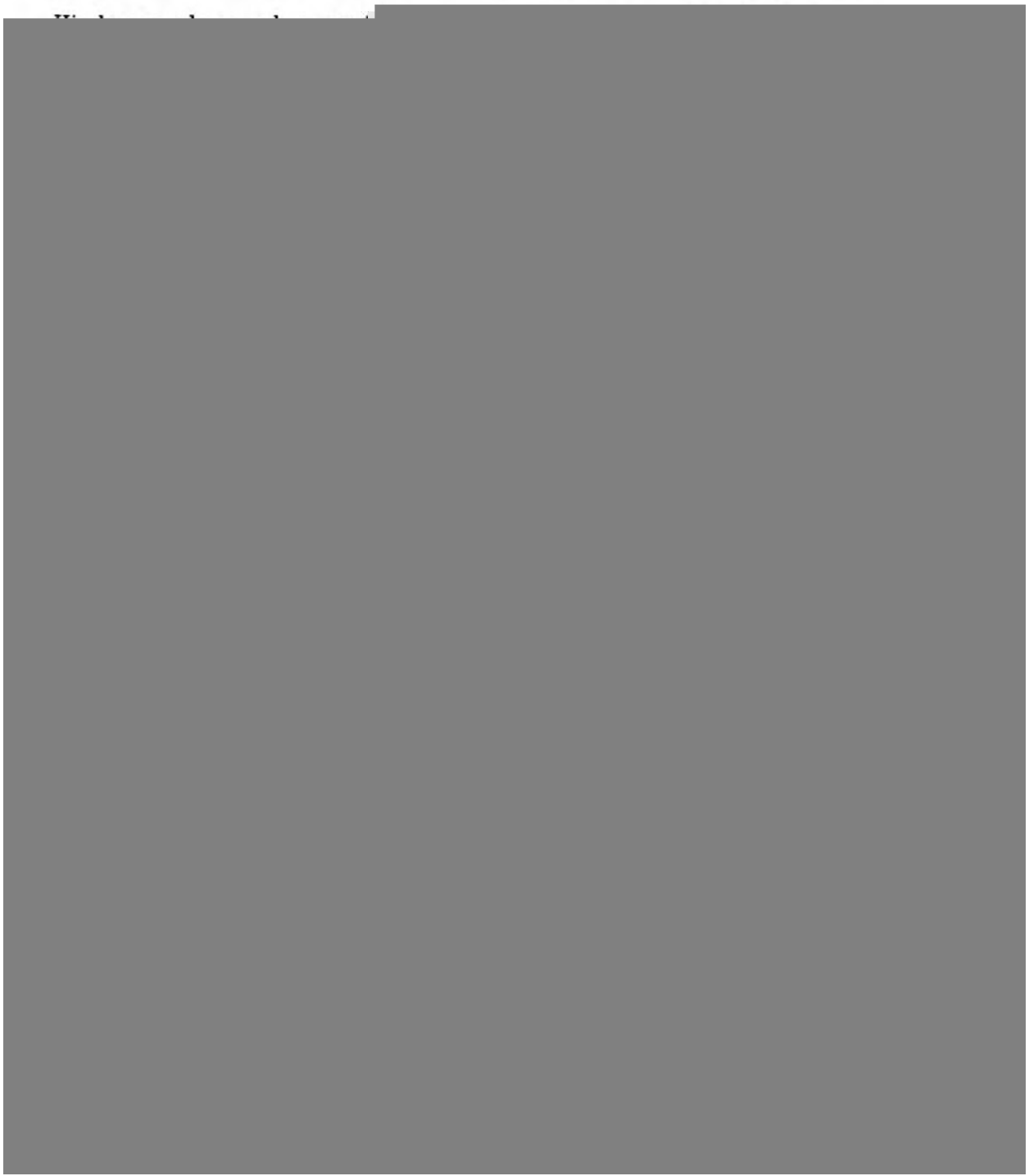
Specific Gravity at 15.5°	0.885 to 0.895
Optical Rotation	0° to −3° 0′
Refractive Index	1.468 to 1.472
Total Geraniol Content	80 to 92%
C[illegible]	[illegible]

7. Lack of cleanliness in distillation apparatus and storage tanks.

8. Improper storage of the distilled oil. After distillation, the oil is often permitted to stand unfiltered for weeks. Solubility of an oil decreases with age, particularly if the oil is left exposed to the air.

9. General climatic conditions. Oils produced during the last weeks of an exceptionally dry period and during the first weeks of the rainy season are apt to show a poor solubility.

10. Soil conditions. These also may affect the solubility of the oil.



work, p. 275). However, it is seldom used in the commercial routine analysis of citronella oil.

As regards the detection of added ethyl alcohol or methyl alcohol, details of procedure will be found in Vol. I of this work, pp. 338 and 340.

Pure Java citronella oils are clearly soluble at all dilutions between 1 and 2 volumes of 80 per cent alcohol, but may become opalescent on further dilution. Turbidity or separation of oil droplets on standing indicates the presence of mineral oil fractions or fatty oils. Such oils should be viewed with suspicion and should be tested

Total Production and Exports.—In the last years prior to the occupation by Japanese forces, Java exported these quantities (in gross weight) of citronella oil: [55]

	Kilograms
1938	1,893,944
1939	2,508,734
1940	2,114,076
1941	2,412,000 (net weight)

Of these three years, 1938 may be chosen as the year in which exports abroad were fairly normal:

	Kilograms
United States	648,937
France	436,522

type of citronella oil contains a substantially higher percentage of these components than the Ceylon type of oil (derived from *Cymbopogon nardus* Rendle, the so-called "lenabatu"), and is therefore more valuable. It also has a stronger odor. Otherwise the two oils are similar except, perhaps, for the content of methyl eugenol, which amounts to more than 8 per cent in the Ceylon oil, and to less than 1

n-1-Hexanol. In the fraction b_{12} 60°–67° of the above-mentioned alcohol mixture b_{15} about 45°–80°, isolated from the foreruns of Java citronella oil, Bohnsack [60] also identified *n*-1-hexanol b_8 53°–55° (α-naphthylurethane m. 60°–61°), after he had first removed the unsaturated components by shaking with a cold solution of potassium permanganate.

Methyl Heptenone. In the course of his work on the foreruns of Java citronella oil

l-Limonene and Dipentene. As regards the terpenes, Chiris [65] identified *l*-limonene and dipentene through their nitrolpiperidines m. 109°–110°, and m. 150°–152°, respectively.

Citronellal, *d*-Citronellol, and Geraniol. Schimmel & Co.[66] found that the Java type of citronella oil contains larger quantities of these chief constituents than the Ceylon oil. According to Gildemeister and Hoffmann,[67] the citronellal content of Java citronella oils ranges from 25 to 54 per cent; in normal oils it is not less than 35

Cadinol. The other of the two sesquiterpene alcohols described by Glichitch [75] and mentioned above was bicyclic (b_3 134°–135°, α_D^{18} −12° 12′). It could not be obtained in crystalline form, but on heating with concentrated formic acid it yielded a sesquiterpene, which gave two derivatives m. 118.5°–119° and m. 123°–124°, identical with cadinene dihydrochloride and cadinene dihydr

Use.—Oil of citronella, Java type, is one of the most important esse

produced in Java, as well as upon the continuance of the supply of genuine, unadulterated lots from Formosa itself. Under Japanese rule and control, the quality of the Formosa oil was always excellent and enjoyed a very

A fully matured plant grows as high as 1.7 m., while the stump attains a

citronella is quite exhausted and, as was pointed out above, fertilizers are not used for reasons of economy.

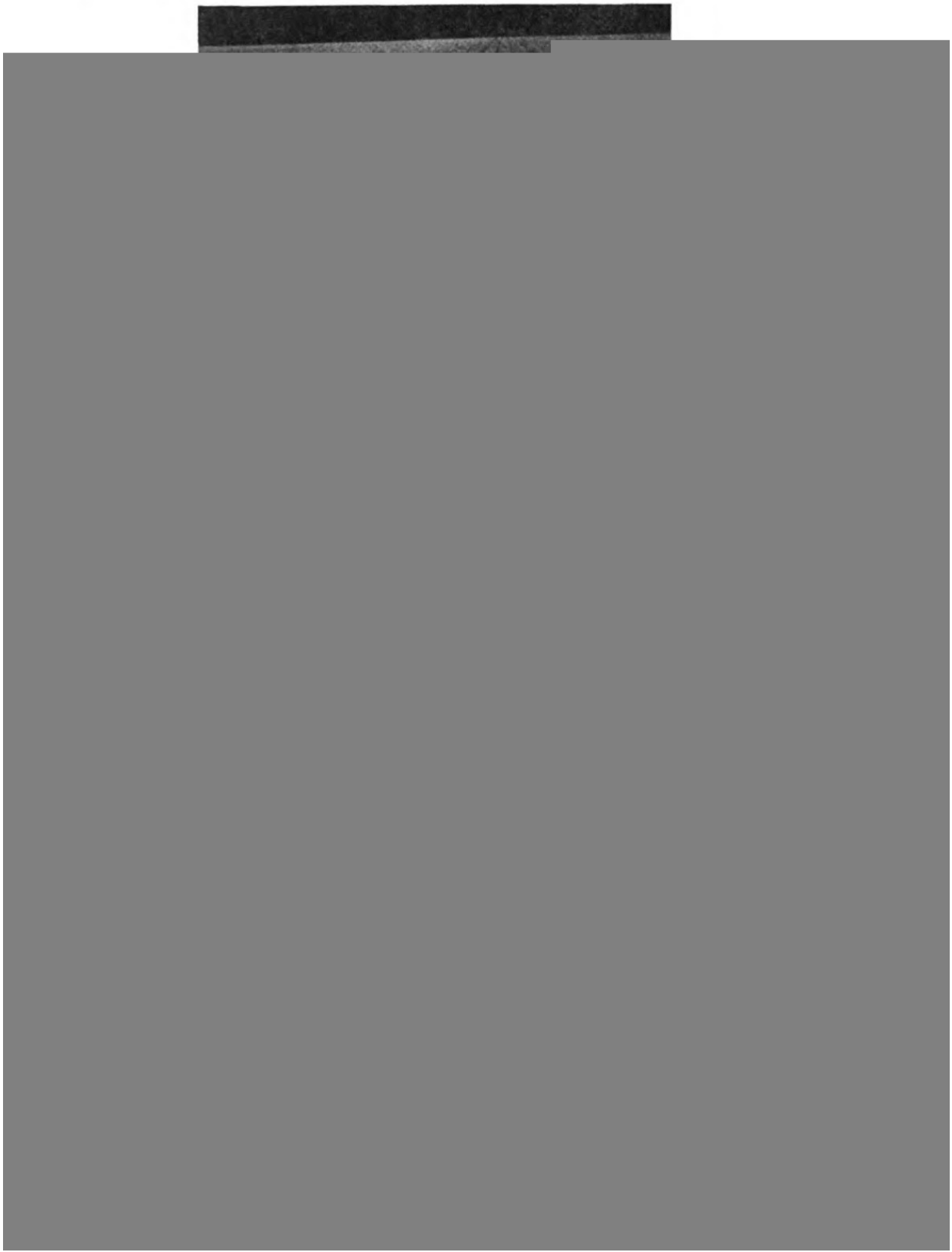
Total Production.—The

Aldehyde Content, Calculated as Citronellal	45.0%
Solubility at 20°	Soluble in 1 vol. of 80% alcohol; opalescent in 4 vol. and more

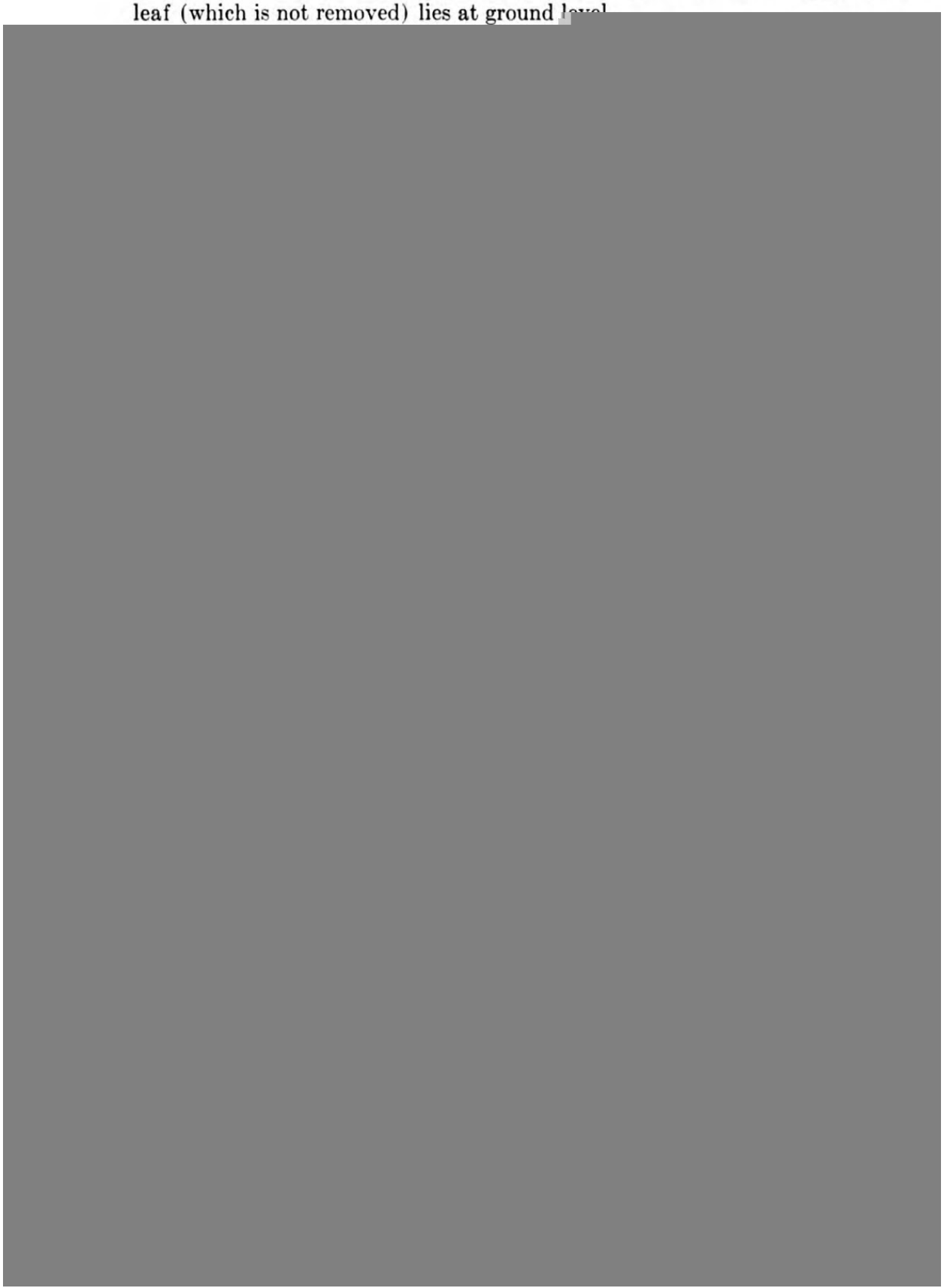
In general, the properties of Formosa citronella oil resemble

Minor Keilhauer that production of oils of citronella and lemongrass could be continued until 1940. The outbreak of World War II brought about a

about 2 ft. long. When preparing the root segments, any large pieces of woody stalk material and dry blades should be cut off and discarded. Three



root segments should be planted in such a way that the base of the lowest leaf (which is not removed) lies at ground level.



In general, it may be said that the yield of oil during the dry season is higher than during the rainy season.

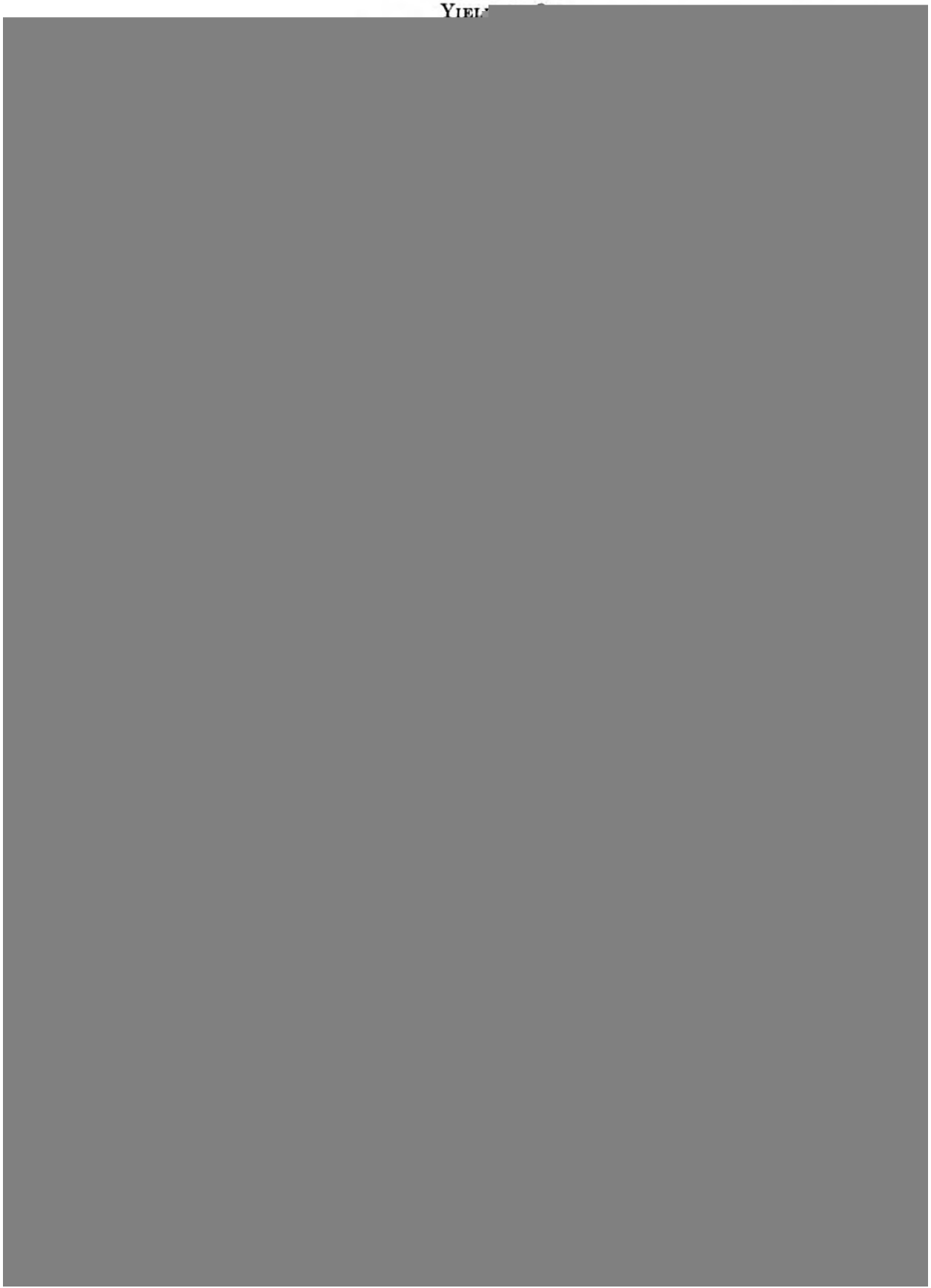
In experiments conducted by Loustalot and Pol,[91] Java citronella grass consistently outyielded the Guatemala grass in all harve

Shipping.—The Guatemala oil is usually shipped to the United States by boat in galvanized iron drums hold[illegible]

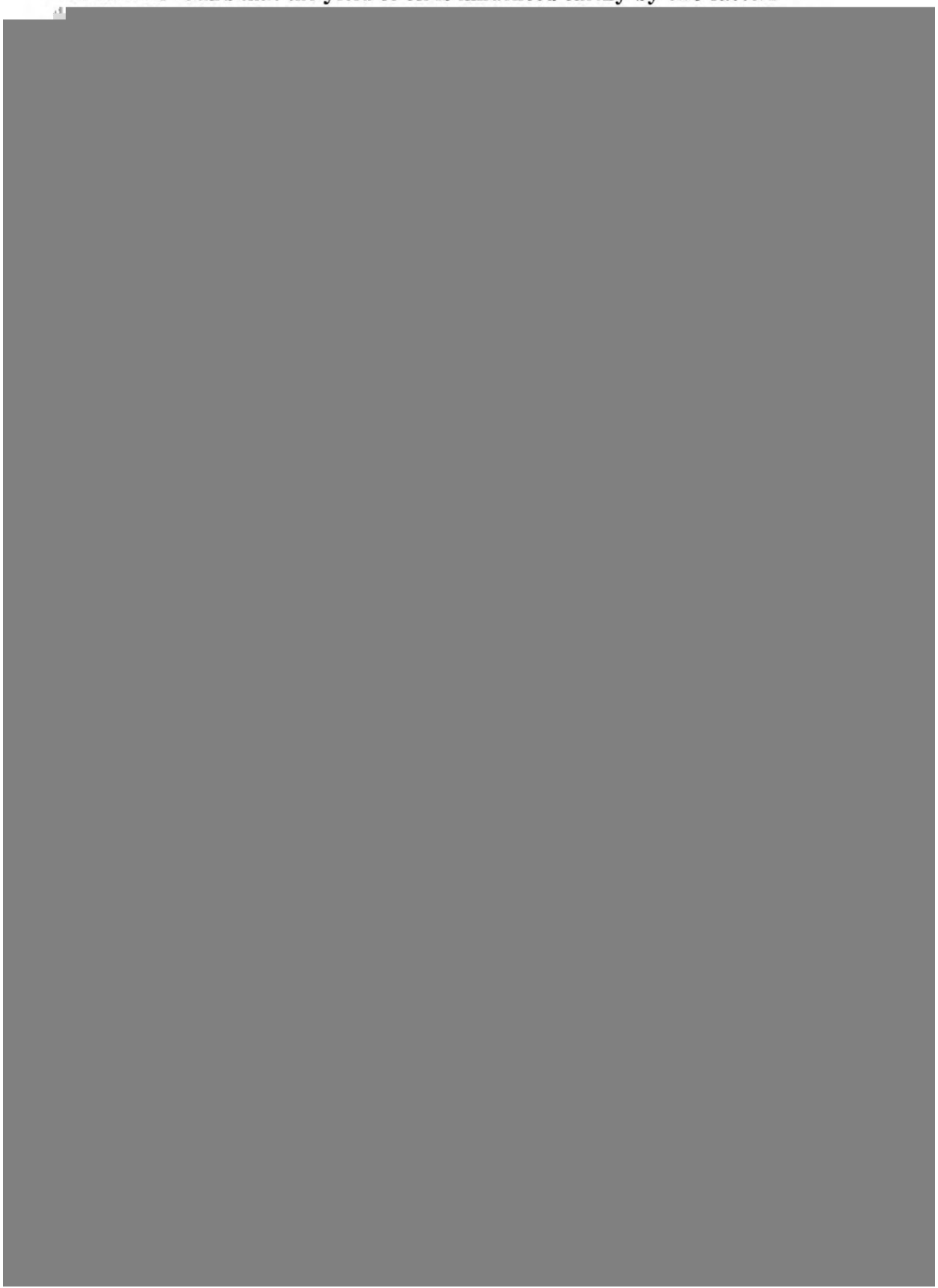
taken to insure purity and to provide oils with the chemical content desired by the purchasers. Important technical and agricultural projects have been

into single or double stalked root segments, which should

Distillation.—Prior to distillation, the leaves are reduced to 4-in. pieces



It has been found that the yield of oil is influenced chiefly by two factors—



Shada (Soc[illegible]

Fiscal Year	*Quantity of Oil in Kilograms*
[redacted]	

Dihydrocuminyl Alcohol (at least 30 to 35 per cent).

An Aldehyde(?) of penetrating odor.

Hydrocarbons(?).

Sesquiterpenic Compounds(?).

Oil of Andropogon Fragrans

Louveau [56] examined a sample of oil of *Andropogon fragrans*, produced experimentally on the island of Réunion. The species had not previously been reported, and Louveau concluded, from the properties and composition of its oil, that the plant might be a third variety of *Andropogon martini* Roxb., of which the *motia* and *sofia* varieties yield palmarosa and gingergrass oils respectively, or possibly a local subvariety of the *sofia*.

The odor of the oil resembled that of palmarosa and gingergrass oils. Properties were as follows:

Specific Gravity at 15°	0.9572
Optical Rotation	+8° 6′
Refractive Index at 20°	1.4942
Acid Number	12.0
Ester Number	17.5
Ester Number after Acetylation	160.3
Total Geraniol Content	50.1%
Ester Content, Calculated as Geranyl Acetate	6.2%
Free Alcohol Content, Calculated as Geraniol	36.1%
Aldehyde Content, Calculated as Citronellal (Burgess Method)	9.2%
Solubility	Soluble in 0.9 vol. of 80% alcohol

The oil consisted of approximately:

10%	Terpenes (about ¼ α-pinene)
6%	Esters of terpene alcohols (chiefly geranyl acetate)
36%	Free alcohols (⅔ geraniol; linaloöl, and perhaps citronellol and nerol)
9%	Aldehydes (¾ citronellal; perhaps citral; certainly cuminic aldehyde)
2%	Ketones (particularly methyl heptenone and menthone)
6%	Free solid acids (chiefly abietic acid)
30%	Sesquiterpenes, resinous substances from polymerization, which did not distill over at b_4 155°.

Oil of Andropogon Intermedius R. Br.

Andropogon intermedius R. Br. (= *Amphilophis intermedia* Stapf) occurs in India, the East Indian Archipelago, Indo-China, China, in the Philippine

[56] *Rev. Marques parfumerie Savonnerie* **11** (1933), 311.

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Islands, and Australia. Occasionally the plant grows in large quantities. In Java the plant is known as "Roempot pipit," in Flores as "Poang." The odor of the oil resembles that of vetiver.

A sample investigated by van Eerde [57] had these properties:

Specific Gravity at 20°	0.9850
Optical Rotation at 20°	−9° 30′
Refractive Index at 20°	1.5018
Acid Number	15.65
Saponification Number	72.64
Saponification Number after Acetylation	176.64
Alcohol Content, Calculated as Geraniol	36.15%
Combined Alcohols	15.68%

The oil contained *l*-limonene, acetic, and butyric acids.

Two samples of oil of *Andropogon intermedius* (R. Br. ?) examined in the Botanical Institute of Buitenzorg [58] (Java) had the following properties:

	I	*II*
Specific Gravity at 26°	0.889	0.919
Optical Rotation	−21° 52′	−15° 30′

The yield of oil in the case of the second sample was 0.03 per cent.

Oil of Andropogon Intermedius R. Br. var. Punctatus Hack.

The oil of *Andropogon intermedius* R. Br. var. *punctatus* Hack., known as "Montsukigaya," was investigated by Kafuku, Sebe and Kato.[59] Obtained in a yield of 0.5 per cent it had the following properties:

Specific Gravity at 20°/4°	0.8600
Optical Rotation at 30°	−10° 0′
Refractive Index at 30°	1.4643
Ester Number after Acetylation	130.18

The oil contained 4-undecanone, a dextrorotatory alcohol, an isomer of decyl alcohol, and octyl alcohol. Content of acids after hydrolysis of the esters reached 7 per cent. Among the acids were acetic, butyric, caproic, enanthic and caprylic acids.

Oil of Andropogon Kuntzeanus Hack. var. Foveolata Hack.

By steam distillation of *Andropogon kuntzeanus* Hack., var. *foveolata* Hack., originating near Poona, in India, Rao [60] obtained 0.27 per cent of an

[57] "Nieuwe Bijdrage tot de Kennis der indische Grasolien," Leyden (1924). Gildemeister and Hoffmann. "Die Ätherischen Öle," 3d Ed., Vol. II, 386.

[58] *Jaarboek van het Departement van Landbouw Nijverheid en Handel in Nederlandsch-Indië*, 1907 (Batavia, 1908), 67; 1910 (Batavia, 1911), 48.

[59] *J. Chem. Soc. Japan* **59** (1938), 312. *Chem. Abstracts* **32** (1938), 8697.

[60] *J. Indian Inst. Science* **15A** (1932), 75.

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Total Production.—During the last ten years (exclusive of the time when Java was occupied by Japanese forces) the following quantities of vetiver oil in metric tons and gross weight were exported from Java: [4]

1939	34
1940	29
1941	23
1947	2
1948	1

Physicochemical Properties.—Four genuine samples of vetiver oil procured by the author while in Java had the following properties:

	I	*II*	*III*	*IV*
Specific Gravity at 15°	1.009	1.009	1.007	0.991
Optical Rotation	+31° 36′	Too dark	+30° 48′	Too dark
Refractive Index at 20°	1.5260	1.5271	1.5260	1.5258
Acid Number	31.1	32.5	30.2	12.5
Ester Number	2.0	12.6	1.0	4.0
Ester Number after Acetylation	141.5	150	152.1	129
Solubility at 20° in 80% Alcohol	Soluble in 1 vol.; cloudy in 2 vol. and more	Not clearly soluble; but soluble in 0.5 vol. of 90% alcohol and more	Soluble in 1.5 vol.; turbid in 3.5 vol. and more	Very cloudy in 10 vol.

Sample IV was inferior to the other oils in regard to odor as well as to properties.

According to Koolhaas and Rowaan [5] (Buitenzorg Government Laboratories) the properties of Java oil vary within these limits:

Specific Gravity at 15°	0.985 to 1.045
Optical Rotation (10 mm. tube)	+15° 0′ to +45° 0′
Refractive Index at 20°	1.510 to 1.530
Acid Number	8 to 35
Ester Number	5 to 25
Ester Number after Acetylation	100 to 150
Solubility in 90% Alcohol	Soluble in 1 to 10 vol.

Shipments of Java vetiver oil received and examined by Fritzsche Brothers, Inc., New York, had properties which varied within the following limits:

Specific Gravity at 15°/15°	1.002 to 1.003
Optical Rotation	+24° 20′ to +31° 4′

[4] Courtesy of Dr. J. A. Nijholt, Laboratorium Voor Scheikundig Onderzoek, Buitenzorg, Java.

[5] *Parfums France* **15** (1937), 245, and private communication by Dr. D. R. Koolhaas

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Refractive Index at 20°	1.5259 to 1.5260
Acid Number	20 to 33
Ester Number	5.6 to 7.5
Ester Number after Acetylation	137.2 to 140.0
Solubility	Soluble in 0.5 vol. and more of 90% alcohol

Gildemeister and Hoffmann[6] distinguished between commercial Java oils of good quality (I), and the so-called "light" oils from Java (II):

	I	*II*
Specific Gravity at 15°	0.9926–1.0444	0.9852–1.0015
Optical Rotation	+20° 30′ to +46° 0′	+14° 25′ to +24° 10′
Refractive Index at 20°	1.51889–1.53060	1.52236–1.52612
Acid Number	8.4–40.1	7.5–14.9; in one case 27.1
Ester Number	5.6–24.6; in one case 2.8	6.5–14.9
Ester Number after Acetylation	103.7–151.2	98–119.5
Solubility in 80% Alcohol	Soluble in 1 to 2 vol.; occasionally opalescent to turbid with more alcohol	Not clearly soluble. Clearly miscible with 90% alcohol; in exceptional cases opalescent on dilution

European-Distilled Vetiver Oil

As was pointed out in the introduction, substantial quantities of baled vetiver root were at one time exported from Java and processed for the recovery of oil in the well-equipped distilleries of Europe. It should be mentioned that, due to shipping difficulties, Réunion Island never exported vetiver root as such. All European-distilled vetiver oils therefore originated from imported Java root.

Vetiver oils distilled under the author's supervision in Southern France had these properties:

Specific Gravity at 15°	1.010 to 1.030
Optical Rotation	+28° 0′ to +40° 0′; usually +33° 0′ to +38° 0′
Refractive Index at 20°	1.5230 to 1.5270
Acid Number	20 to 33
Ester Number	6 to 10
Ester Number after Acetylation	145 to 170
Solubility in 80% Alcohol, at 20°	Usually soluble in 0.5 to 1 vol.; occasionally opalescent and slightly turbid on addition of more alcohol

Owing to the modern apparatus and methods of distillation applied, these oils were of very high quality.

[6] "Die Ätherischen Öle," 3d Ed., Vol. II, 329.

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a season's growth usually persist on the branches for about two years, but some may be retained for five or six years.

To collect the leaf and branch material, long-arm pruning clippers capable of clipping branches more than an inch thick should be used. By this method most of the trees of average height growing in California can be pruned. Packing of stills with branches over 15 mm. thick is inconvenient.

Yield of Oil.—According to Swanee,[3] the yield of oil varies and depends on the season and location. The yield was found to be greatest in summer and fall. The leaves and stems (stems not over 5 mm. thick) yield as high as 4.1 per cent of oil. The stems alone yield from 0.56 to 1.13 per cent; the leaves alone as much as 4.69 per cent of oil. The average of seven distillations of leaves and stems was 2.87 per cent.

Physicochemical Properties.—The oil possesses an extremely pungent odor and is strongly lachrymose, causing severe headache on inhalation. The oil has, therefore, to be handled with great care. The flavor is warm and camphoraceous.

Gildemeister and Hoffmann [4] reported the following physicochemical properties for several California bay oils:

Specific Gravity at 15°	0.916 to 0.948
Optical Rotation	−22° 0′ to −24° 0′
Refractive Index at 20°	1.47210
Acid Number	0.4 and 4.7
Ester Number	5.5 and 5.6
Ester Number after Acetylation	29.9 and 50.8
Solubility	Soluble in 1.5 to 2.2 vol. of 70% alcohol; soluble in 1 vol. and more of 80% alcohol. The diluted solutions are occasionally opalescent

Shipments of genuine California bay oils received and examined by Fritzsche Brothers, Inc., New York, had properties varying within these limits:

Specific Gravity at 15°/15°	0.927 to 0.934
Optical Rotation	−26° 13′ to −28° 20′
Refractive Index at 20°	1.4750 to 1.4769
Ketone Content, Calculated as Umbellulone (Sodium Sulfite Method)	40 to 43%
Solubility	Soluble in 1.5 to 2 vol. and more of 70% alcohol

[3] *Ibid.*, December (1944), 82.
[4] "Die Ätherischen Öle," 3d Ed., Vol. II, 710.

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Eliminating the first fractions (b. up to 155°, or about 8 to 10 per cent of the oil), Swanee [5] found that the remaining oil had a pleasant odor.

Chemical Composition.—The first investigations of the chemical composition of the volatile oil derived from the California bay tree were carried out by Heaney,[6] and by Stillmann,[7] but were rather inconclusive. Later, Power and Lees,[8] Schimmel & Co.,[9] Semmler,[10] and Lees [11] and Tutin [12] succeeded in elucidating not only the chemical composition of the oil, but also the structure of its chief constituent, viz., umbellulone, a ketone, $C_{10}H_{14}O$, of very pungent, mint-like odor.

According to Power and Lees,[13] the oil contains the following compounds:

Eugenol. In small quantities; identified by means of its benzoyl compound m. 69°–70°.

l-α-Pinene. Identified by preparation of its nitrolpiperidine m. 119°–120°, and nitrolbenzylamine m. 124°–125°.

Cineole. Iodol compound m. 115° (Schimmel & Co.[14]).

Safrole. Very small quantities. Characterized by oxidation to piperonal m. 35° (Power and Lees).

Methyl Eugenol. Identified by means of the bromoeugenol methyl ether dibromide m. 78°–79°, and by oxidation to veratric acid m. 177° (Power and Lees).

Formic Acid and Higher Fatty Acids. The oil contains traces of formic acid and higher fatty acids in free form (Power and Lees).

Umbellulone. The chief component of the oil, umbellulone, is present in amounts ranging from 40 to 60 per cent (Power and Lees).

Regenerating the substance from its semicarbazido-semicarbazone m. above 200°, Semmler [15] obtained the pure ketone $C_{10}H_{14}O$, which had these properties: b_{10} 92.5°–93°, d_{20} 0.950, α_D −36° 30′, n_D 1.48325. Power and Lees [16] reported b_{749} 219°–220°.

For further details and the structural formula, the reader is referred to Vol. II, p. 440, of this work.

[5] Conference on the Cultivation of Drug and Associated Economic Plants in California, Proceedings (1944), 82. (California State Department of Education and California Polytechnic School, San Luis Obispo, Calif.)
[6] *Am. J. Pharm.* **47** (1875), 105. *Pharm. J.* [3] **5** (1875), 791.
[7] *Ber.* **13** (1880), 630.
[8] *J. Chem. Soc.* **85** (1904), 629.
[9] *Ber. Schimmel & Co.*, October (1893), Table in Appendix, 27.
[10] *Ber.* **40** (1907), 5017; **41** (1908), 3988.
[11] *J. Chem. Soc.* **85** (1904), 644.
[12] *Ibid.* **89** (1906), 1104; **91** (1907), 271, 275; **93** (1908), 252.
[13] *Ibid.* **85** (1904), 629.
[14] *Ber. Schimmel & Co.*, October (1893), Table in Appendix, 27.
[15] *Ber.* **40** (1907), 5017; **41** (1908), 3988.
[16] *J. Chem. Soc.* **85** (1904), 629.

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OIL OF MASSOI BARK

There has been a great deal of confusion regarding the taxonomy of the plant, the bark of which, on steam distillation, yields oil of massoi. It was only in 1937 that Abe [1] succeeded in clarifying the problem of the origin and the properties of true massoi bark oil. The most recent and thorough investigation has been carried out by Meijer.[2]

Meijer showed that in a number of cases where oil of massoi bark has been described in literature, the description actually refers to the oil of lawang bark. Oil of massoi bark does not contain eugenol, whereas oil of lawang bark contains a considerable amount. The chief constituent of massoi bark oil, massoilactone, was first isolated by Abe, but its correct structure was established by Meijer [3] as a lactone $C_{10}H_{16}O_2$, details of which will be found in Vol. II of this work, p. 688.

According to Meijer, the principal oil-containing barks of Netherlands New Guinea are those of massoi and lawang. Massoi bark has been collected for centuries. Trade in the bark was once of such importance that, in the seventeenth century, the East India Company organized special expeditions to New Guinea to procure the bark.

The latter, handled in bundles weighing about 10 kg., consists of pieces about 100 cm. in length and 5 cm. in width. It has some medicinal value and forms an ingredient of Javanese medicines, called obat or djamoe, which consist of a mixture of various herbs.

The botanical name of the plant from which true massoi bark is derived had been a matter of much controversy until Salverda [4] made an expedition to New Guinea and collected several authentic specimens of the bark. These were identified as *Cryptocaria massoia* (fam. *Lauraceae*) at the government station in Buitenzorg. Previously Gildemeister and Hoffmann [5] had reported several names for the plant, viz., *Massoia aromatica* Beccari, *Cinnamomum xanthoneuron* Blume, and *Cinnamomum culilawan* Blume.

A great deal of misunderstanding has occurred in the description of lawang and massoi oils. Very often literature refers to a massoi oil where, according to physicochemical properties and constituents, the reference should be

[1] *J. Chem. Soc. Japan* **58** (1937), 246.
[2] *Rec. trav. chim.* **59** (1940), 191.
[3] *Ibid.*
[4] *Rapport van een oriënteerende exploratie in Z. W. Nw. Guinea* van Nov. 1937 tot Mei 1938, 93. See also *De Bergcultures* **13** (1939), 1265. Cf. Meijer, *Rec. trav. chim.* **59** (1940), 192.
[5] "Die Ätherischen Öle," 3d Ed., Vol. II, 714.

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In their quest for spices, early Portuguese explorers rounded the Cape of Good Hope and, conquering Ceylon, forced the Singhalese kings to pay tribute in the form of cinnamon bark collected from wild-growing trees. About 1770, during their rule over the island, the Hollanders started to plant cinnamon and established a strict control over the spice by regulating production and destroying any surplus in order to keep prices high. Taking advantage of Holland's embroilment in the Napoleonic wars, England took over Ceylon in 1796, and its cinnamon industry became a monopoly of the powerful East India Company which lasted until 1833.

Botanical Origin.—There has been much confusion, in the trade as well as in official standard texts, in regard to the botanical origin of cinnamon bark, and the oil distilled from it. In fact, in some cases it is almost impossible to establish to what particular botanical species or varieties certain types of cinnamon bark belong. Climatic and soil conditions affect the Ceylon cinnamon plant so profoundly that the bush or tree, when grown in other countries, produces a bark quite different from that of the Ceylon-grown bush. Moreover, the appearance and quality of the bark depend upon the age of the tree, the method of collection and preparation. For example, the dried *inner* bark of the shoots of *coppiced bushes* (true Ceylon cinnamon) differs substantially from the dried *whole* bark of *semiwild trees* (Madagascar cinnamon), although the Madagascar cinnamon plant was originally introduced from Ceylon. Similar conditions apply to the Seychelles cinnamon.

Official standard works describe three principal types of cinnamon, but in regard to the volatile oils do not always clearly differentiate between them.

1. Ceylon cinnamon. The National Formulary, Eighth Edition, describes as Ceylon cinnamon the dried inner bark of the shoots of coppiced trees of *Cinnamomum zeylanicum* Nees (fam. *Lauraceae*). The British Pharmacopoeia of 1948 recognizes the same drug under the collective term cinnamon, and specifies that the essential oil distilled from it contain not less than 50 per cent, and not more than 65 per cent, by weight, of cinnamic aldehyde. The British Pharmacopoeia of 1948 does not list cassia or cassia oil.

The present monograph will deal chiefly with Ceylon and closely related cinnamon oils.

2. Saigon cinnamon. The Saigon cinnamon tree is of medium height, originally native to China. Its bark is collected chiefly in the mountains of Anam (French Indo-China) from semiwild trees. Most of the bark comes from branches or from the trunks of young trees, but chips of thicker bark are occasionally included. The principal shipping port is Saigon, although

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tities of cinnamon, the best grades of which were exported largely to Spain. The Spanish civil war (1936–1939) and economic and political developments in general have changed the picture, and many of the cinnamon plantings in the Negombo district are now replaced with coconut. (2) The regions south of Colombo, stretching along the seashore, with many small cinnamon plantings. The main centers here are Ambalangoda, Galle, and Matara in the far south of the island. The bulk of cinnamon originates from here, but the quills, probably because of the harder gravel soil, are somewhat thicker and coarser than those from the Negombo district.

In general, cinnamon plantations are scattered along Ceylon's western seacoast, from Negombo all the way down to Matara. The planters, as well as the local dealers, are for the most part Singhalese, who own estates of from 2 to 300 acres, but mostly small holdings of less than 20 acres. For the last twenty-five years, the total plantings have averaged 25,000 acres. As was said, the cinnamon plantings in the south have expanded, while those in the north have been partly abandoned. This movement from north to south is still going on, Matara with its suitable soil becoming an increasingly important center. The acreage of cinnamon plantings directly reflects the market price of the quills, as compared with prices of other Ceylon produce. Recently, rubber and coconut have made large inroads into the acreage formerly devoted to cinnamon.

Aside from the planted cinnamon, there is also a very small amount of wild cinnamon being produced in Ceylon, and used almost exclusively for local consumption. It is difficult to prepare quills from the thick bark of wild growing cinnamon trees, but the native dealers sometimes mix chips of wild cinnamon with those of planted cinnamon, even though this practice is prohibited, as a fraudulent manipulation. Experts can identify wild cinnamon by its appearance and by its somewhat acid and comparatively weak taste. It originates from the Ratnapura and Badulla districts in the southwestern parts of Ceylon, where it grows spontaneously in the jungles, being propagated by birds.

Planting, Cultivating, and Harvesting.—To obtain seed, the fruit on the bushes is either protected with bags against birds, or collected before ripe, after which it is left to rot in the shade until the clean seed can be removed from the mass of pulp by trampling. The washed and shade-dried seed is planted in a nursery (about 8 seeds in one hole), well watered and shaded. After six or seven months, the young plants are ready for transplanting. If a nursery bed is not available, the seedlings are purchased from a neighboring plantation and then set out 6 or 7 ft. apart on virgin land. After about three years, during which they have to be pruned to grow straight, the young stems are cut down to a few inches aboveground. They are sometimes covered with earth to facilitate the development of stools, on which

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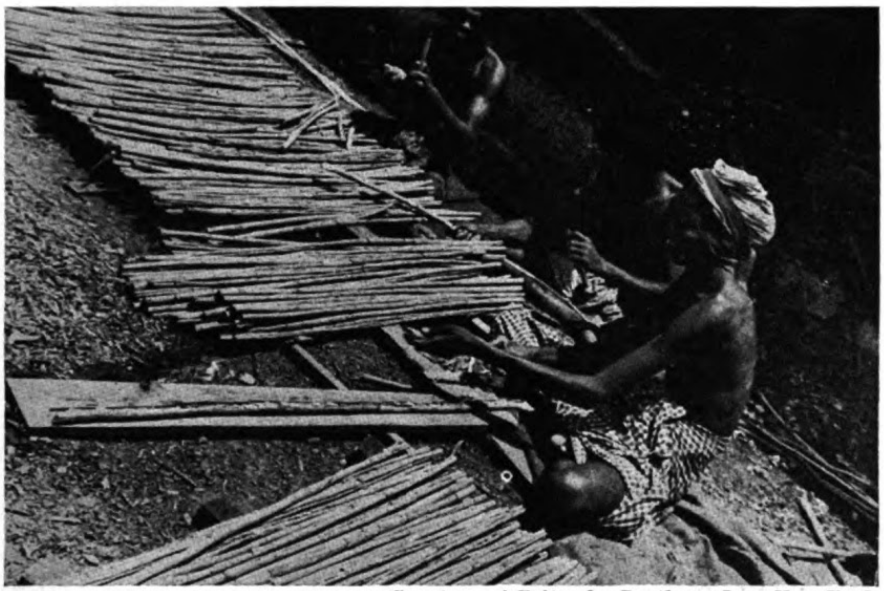
Ceylon cinnamon bush, of which the outer coating has been removed. This inner bark, which turns yellow-brown upon drying, contains the essential oil and the flavoring principles. The external coating has a weak and slightly acid flavor, the absence of which seems to account for the fine flavor for which true Ceylon cinnamon is so much appreciated. The same applies to the essential oil distilled from Ceylon cinnamon bark. The quills, under which form Ceylon cinnamon is marketed, consist of two or more dried



Courtesy of Fritzsche Brothers, Inc., New York.

FIG. 20. Cinnamon production in Ceylon. Preparation of cinnamon quills.

tubes of inner bark which are tightly rolled together, after the hollow interior has been filled with small pieces of broken bark. Superior grades, however, should not contain any fillings of broken pieces. They are prepared as follows:

After the freshly cut branches of the cinnamon bush arrive at the working place, the leaf material is removed with bush knives, and the woody, inert external bark is scraped off with curved knives, great skill being required not to scrape or cut into the valuable inner bark. The removed outer bark may be used for green fertilizing. The longest straight stalks are selected to make the best quills. After the outer bark has been removed, a longitudinal incision, 6 to 8 in. long, is made with a sharp knife into the soft, white, inner bark, which is then very carefully and deftly peeled off the hard core. Thus, hollow tubes 6 or 8 in. long are obtained, into which smaller or broken pieces are packed. Finally, several of the 6 or 8 in. quills are telescoped together to form tubes about 36 in. long. These are rolled by hand while fresh and

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soft, and very slightly pressed. Obviously, native producers are tempted to fill the hollow tubes with inert foreign material, such as wood, sand, and earth, instead of true cinnamon. However, careful exporters, aware of the possibility of such a fraud, examine the quills when buying from the natives, before they make shipments abroad. It is, therefore, advisable for the consumer abroad to buy cinnamon quills only from reliable shippers who will give full guarantee for purity.

The rolled quills are laid on mats and dried for three days in the sun and for three days in the shade. The quills must be rolled by hand and slightly pressed every day during the drying process, because otherwise they will swell up and split. In the course of the drying operation, the originally white color of the quills slowly turns darker and assumes a yellow-brown tint. Drying also gives the quills the appearance of fairly strong sticks.

Yield per Acre.—One acre planted with cinnamon yields about 1.5 or 2 bales (a bale contains 100 lb.) of cinnamon quills, and, in addition, ½ cwt. of cinnamon chips and 15 to 20 cwt. of fresh, undried cinnamon leaves.

Commerce.—As a rule, the growers do not sell their cinnamon directly to exporters, but transport it to the nearest buying centers (Colombo, Ambalangoda, or Matara), where native dealers collect the bundles, bleach the quills if necessary, assort them into different qualities, and finally make them up into bales of approximately 100 lb. net. These dealers then enter contracts with the exporters in Colombo for 100 or more bales at a time.

Quality of the Cinnamon.—The quality and appearance of assorted cinnamon naturally depend a great deal upon the region of production and the care exercised in its preparation.

(A) Quills:

The quills, which actually consist of compound quills, usually have a length of about 1 m. and a thickness up to 1 cm. or more. They are pale brownish-yellow in color, and thin, brittle, and smooth in texture. They should contain no cork, except for a little around the scars, where the lateral branches have been removed. Every exporter has his own standards (which, however, vary but little). If deliveries are up to standard, no further work in respect to quality is required in the exporters' stores. One of the best known Colombo shippers, for instance, markets the quills under the following designations:

(1) "Fines" 00000 0000 000 00 0	00000 is the finest grade, 0 the coarsest. Best cinnamon is produced largely to the north of Colombo, near Negombo.
(2) "Barcelona" or "Spanish" I II III	A medium quality. B-I is slightly thicker than 0. B-II is about equal to "Hamburg" I. B-III consists of thin but "foxy" (i.e., reddish) quills, which have to be sorted out of

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These properties differ somewhat from those of European or American distilled *bark* (quills) oils, the properties of the oil distilled from chips showing the greatest deviation. Because of its very high eugenol content, the latter is almost in a class by itself, standing between bark oil and leaf oil. Yet this oil, too, had a very good odor and flavor; in fact, the eugenol seemed to bring out a characteristic note esteemed by many connoisseurs.

A critical examination of the properties in general reveals that the eugenol content and specific gravity increase, while the cinnamic aldehyde content, refractive index, and the laevorotation decrease, as the raw material is changed from quillings to featherings and finally to chips.

Native-distilled cinnamon oils imported and examined by Fritzsche Brothers, Inc., New York, had properties varying within the following limits:

Specific Gravity at 15°/15°	1.020 to 1.030
Optical Rotation	−1° 0′ to −2° 10′
Refractive Index at 20°	1.5680 to 1.5700
Aldehyde Content, Calculated as Cinnamic Aldehyde (Hydroxylamine Hydrochloride Method)	51.8 to 56%
Phenol (Eugenol) Content	14 to 18%
Solubility at 20°	Soluble in 1.5 to 2.5 vol. of 70% alcohol, occasionally opalescent to hazy with more

The properties of cinnamon oils distilled by Fritzsche Brothers, Inc., New York, from high-grade Ceylon cinnamon quills ("Fines" 0000) have varied within these limits:

Specific Gravity at 15°/15°	1.011 to 1.1030
Optical Rotation	−0° 6′ to −0° 52′
Refractive Index at 20°	1.5748 to 1.5870
Aldehyde Content, Calculated as Cinnamic Aldehyde (Hydroxylamine Hydrochloride Method)	62.2 to 75%
Phenol (Eugenol) Content	6 to 13.2%
Solubility	Soluble in 2 to 3 and more volumes of 70% alcohol; occasionally with opalescence

These oils were of light yellow color; they possessed the delicate odor of true Ceylon cinnamon and a characteristic spicy, sweet, and warm flavor.

In a few cases Fritzsche Brothers, Inc., obtained oils of lower cinnamic aldehyde content, which had a correspondingly lower specific gravity and refractive index.

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Variety "B"

Citral. The oil of variety "B" contains from 16 to 20 per cent of citral d_{15}^{15} 0.8928, n_D^{20} 1.4883. The citral yielded two semicarbazones m. 163°–164°, and m. 135°–136°, respectively.

Geraniol (Free and as Formate and Acetate). Identified by means of the silver salt of the phthalic acid ester m. 133°, and of the diphenylurethane m. 82°–83°. The geraniol d_{15}^{15} 0.880, α_D^{20} +1° 26′, n_D^{20} 1.4699 occurs in the oil both free and as formate and acetate.

Citronellol. Citronellol is also present in free and in ester form.

Oil of Melaleuca Alternifolia Cheel

Botany.—This species was raised to specific rank by Cheel.[10] It had previously been described by Maiden and Betche[11] as a variety of *Melaleuca linariifolia*. The tree bears a superficial resemblance to *Melaleuca linariifolia*, but is distinct botanically. Both trees are "Narrow-leaved" paper bark "Tea Trees," but *Melaleuca alternifolia* is not as tall as *M. linariifolia*, the height not exceeding 20 ft. The leaves are alternate, narrower, and usually shorter than those of *M. linariifolia*. The whole plant is glabrous and the flowers are generally scattered in an interrupted spike.

Habitat, Range, and Occurrence.—*Melaleuca alternifolia* follows watercourses and flourishes in swampy situations. The tree occurs in fairly large stands in the Northern Rivers districts of New South Wales, from the southern limit at Stroud, through the coastal rivers to southern Queensland.

Planting and Cultivation.—*Melaleuca alternifolia* responds readily to cultivation from seed, and good results have been obtained in experimental plantations. Notwithstanding the export demand for the oil of *M. alternifolia*, the existing stands have so far met all requirements. However, fairly large areas of country are destroyed from time to time by bush fires.

Collection of Leaf Material.—To collect the leaf material the trees are cut down to within 5 or 6 ft. of the ground, and the limbs are cut off. The leaves are removed with a cane knife, and allowed to drop upon squares of hessian, each holding from 40 to 80 lb. The foliage is then carted to the distillery.

Within eighteen months to two years after cutting of the original trees, new growth, known as "ratoon," makes its appearance. Depending upon climatic conditions, this new growth is ready for cutting within eighteen months to two years.

[10] "Notes on *Melaleuca*, with Descriptions of Two New Species and a New Variety," *J. Proc. Roy. Soc. N. S. Wales* **58** (1924), 189.

[11] *Proc Linnaean Soc. N. S. Wales* **29** (1904), 742.

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Distillation.—Two methods are employed in distilling the leaves of this species—namely, distillation in directly fired stills, and distillation of the foliage with live steam generated in a separate boiler. The stills used for direct firing resemble those described in the monograph on "Australian Eucalyptus Oils," in the section on "Production." Made of mild steel, and holding from 1½ to 2 tons of leaves, they are connected to a water tank of approximately 1,500 gal. capacity, in which the condenser coil for cooling of vapors is placed. The distillation occupies about 6½ hr.

The large plants using live steam employ boilers ranging from 24 to 40 h.p. The stills used in this operation are slightly smaller than those employed for direct firing, as they do not require a water space in the bottom of the stills. A wire tray to support the foliage rests immediately on top of the steam pipes, and live steam of about 15 to 20 lb. pressure is passed through the leaves. The oil and steam are condensed in the same manner as in the case of the directly fired still. The methods of packing and emptying the still are essentially the same, and there is no difference in the yield and quality of the oil.

The live steam unit consists of a steam boiler with three stills, each with its separate condenser tank. Overhead derricks or endless chain blocks are used to empty the stills rapidly.

Directly fired stills are used where the stands of *Melaleuca alternifolia* are not sufficiently large to warrant steady all-year-round operation of a still. In order to keep a large steam plant busy it is necessary to have sufficient stands of the tea tree within a radius of about 12 miles of the distillery.

Yield of Oil.—One thousand pounds of leaves and terminal branchlets yield about 18 lb. of a pale lemon-tinted oil possessing a pleasant nutmeg odor. The yield of oil is lower in the winter months than in the summer, a sudden increase appearing in November, the first month of summer. The yield declines again about June, the first winter month in Northern New South Wales.

As regards the cineole content of the oil, an increase appears to occur in the winter months, corresponding to the lowered yield.

Physicochemical Properties.[12]—

Specific Gravity at 15°/15°	0.8950 to 0.9050
Optical Rotation	+6° 48′ to +9° 48′
Refractive Index at 20°	1.4760 to 1.4810
Ester Number	2 to 7
Ester Number after Acetylation	80 to 90
Cineole Content	Under 10%
Solubility in 80% Alcohol (W/W)	Soluble in 0.6 to 0.8 vol.

[12] Penfold and Morrison, "Australian Tea Trees of Economic Value," *Sydney Technological Museum, Bull.* No 14, Part I, 3d Ed. (1946), 12.

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OIL OF LOVAGE

Essence de Livèche *Aceite Esencial Levistico* *Liebstocköl*
Oleum Levistici

Botanical Description.—*Levisticum officinale* Koch, syn. *Angelica levisticum* Baillon, is a tall perennial, growing under favorable conditions to a height of almost 6 ft. All parts of the plant are strongly aromatic. The roots, which constitute the commercially most important part of the plant, are composed of a vertical rhizome, 5 to 10 cm. long and 2 to 4 cm. thick, from which spring forth numerous lateral rootlets, up to 20 mm. long and a few millimeters thick.

All parts of the plant contain a volatile oil, which can be recovered by steam distillation, the root oil being the most important one.

According to The Herb Society of America,[1] lovage, a once popular but now somewhat neglected herb, is distinguished by its strong celery-like flavor and aroma. It was, indeed, used as a celery substitute by our forefathers. In old New England the root used to be candied in sugar syrup in the same way as sweet flag root. Lovage is easy to grow and adaptable to almost any soil, although it thrives best in a rather moist, heavy soil with a good deal of shade. Here it will grow to a height of 7 to 8 ft. and send up a flower stalk another foot high.

The roots have long been supposed to possess medicinal properties; they are administered in the form of infusions as a stimulant aromatic, and as a carminative, diaphoretic, and emmenagogue. The seeds (fruits) are used for flavoring confectionery and notably in liqueurs of French type. The leaves are sometimes blanched, like celery, and relished in the form of salads.

Habitat and Range.—Lovage is a native of Southern Europe, where it grows wild in the mountain regions of Southern France (Dauphiné and Provence). It thrives best in damp and strong soils. Lovage is cultivated in Germany (Thuringia and Bavaria), also in Hungary, Czechoslovakia, and other central European countries. The perennial has been introduced to the United States as a garden plant and is now grown as a crop in a few localities of New England and the West.

Planting, Cultivating, and Harvesting.—Stockberger [2] recommends propagating lovage by root division or from seeds. The seeds may be planted in the fall in drills 18 in. apart or sown in early spring in a hotbed, green-

[1] *The Herbarist,* No. 10, Boston, Mass. (1944).
[2] "Drug Plants under Cultivation," *U. S. Dept. Agr., Farmers' Bull.* No. 663 (1935), 27.

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house, or well-prepared seedbed, in a sheltered portion of the garden. They should be covered very lightly with sand or fine-sifted soil. To prevent the soil from drying out before the seeds germinate, it is advisable to spread old burlap or sacking over the bed. The sacking may be sprinkled occasionally, if the weather is dry, and should be removed when the first seedlings break the soil. The plants should reach a size suitable for transplanting by the end of May, when they may be set at intervals of 8 in., in rows far enough apart for convenient cultivation. Lovage grows well in almost any deep, well-drained soil, such as will produce a fair crop of corn or potatoes, and is benefited by the liberal use of fertilizer, although heavy application of manure tends to produce excessive top growth.

The roots may be dug in October of the second or third year after setting the plants. Numerous offshoots will generally be found; if these have good roots they may be used to renew the plantings, without recourse to seed. Such shoots should be reset immediately at the usual distances apart. The freshly dug roots should be well washed, cut into slices about one-half inch thick, and carefully dried. If necessary, artificial heat not exceeding 125° F. may be used to hasten drying.

According to Stockberger,[3] returns from experimental areas indicate that under good conditions a yield of about 1,000 lb. of dried root to the acre may be expected every third year.

Distillation of Lovage Root.—Age of the root material affects the quality of the recovered oils, oils from fresh roots being considered superior. Distillation of dried roots gives rise to the formation of a yellow, gummy, and sticky mass in the condenser and oil separator, especially toward the end of operation. The resulting oils are brown in color. Distillation of green, especially of freshly harvested, roots, on the other hand, proceeds without any gum formation; the oils are then of yellowish color. Rectification of oils from old root material leaves as residue considerable quantities of resinous matter, whereas oils from fresh roots are almost entirely volatile.

Yield of Lovage Root Oil.—Distillation of very fresh root material with direct steam yields 0.1 to 0.2 per cent of oil, semidried roots 0.3 to 0.6 per cent, and dried roots 0.6 to 1.0 per cent of oil. It is advisable to granulate the root material prior to distillation; otherwise the yield will be subnormal.

Physicochemical Properties.—According to Gildemeister and Hoffmann,[4] the properties of lovage root oil vary within the following limits:

Specific Gravity at 15°	1.00 to 1.05
Optical Rotation	Inactive to slightly dextrorotatory, up to +6° 0′; seldom laevorotatory, up to −1° 0′

[3] *Ibid.*

[4] "Die Ätherischen Öle," 3d Ed., Vol. III, 540.

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